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FINAL SAMPLING AND ANALYSIS PLAN FOR LONG TERM GROUNDWATER MONITORING
OPERABLE UNIT 1 SITE 1 SANITARY LANDFILL NAS PENSACOLA FL
4/1/2014
RESOLUTION CONSULTANTS

FINAL

SAMPLING AND ANALYSIS PLAN

**LONG-TERM GROUNDWATER MONITORING,
OPERABLE UNIT 1 — SITE 1 SANITARY LANDFILL
NAVAL AIR STATION PENSACOLA
PENSACOLA, FLORIDA**

Version Number: 0

Prepared for:



**Department of the Navy
Naval Facilities Southeast
Building 135 North, P.O. Box 30
Jacksonville, Florida 32212-0030**

Prepared By:



**Resolution Consultants
A Joint Venture of AECOM & EnSafe
1500 Wells Fargo Building
440 Monticello Avenue
Norfolk, Virginia 23510**

**Contract Number: N62470-11-D-8013
CTO JM40**

April 2014

SAP WORKSHEET #1: TITLE AND APPROVAL PAGE

(UFP-QAPP Manual Section 2.1)

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Cantwell.Tina

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Tina Cantwell, Resolution Consultants
Project Chemist/Quality Assurance Officer/Date

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Allison Harris, Resolution Consultants
Task Order Manager/Date

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Quality Assurance Officer/Chemist/Date

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Patty Marajh-Whittemore, NAVFAC Southeast
Remedial Project Manager/Date

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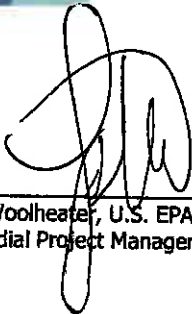
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CTO JM40**

April 2014


Tim Woolheater, U.S. EPA
Remedial Project Manager/Date

4/22/14


David Grabka, FDEP
Remedial Project Manager/Date

4/25/14

EXECUTIVE SUMMARY

This Uniform Federal Policy Sampling and Analysis Plan Sampling and Analysis Plan (UFP-SAP) constitutes the planning document for the Long-Term Monitoring Program for Groundwater and Surface Water at Operable Unit (OU) 1 at Naval Air Station Pensacola, Florida. Resolution Consultants has prepared this SAP under the Comprehensive Long-Term Environmental Action Navy Contract No. N62470-11-D-8013 Contract Task Order JM40.

The overall goal of this SAP is consistent with the previous UFP-SAP completed for OU 1, by Tetra Tech in 2010. Because of a new Navy contractor (Resolution Consultants), changes have been made to document current communication pathways, lines of authority, and standard operating procedures. Updates have also been made to laboratory detection limits as needed to reflect current limits and the Department of Defense Quality Systems Manual.

OU 1 specific changes include:

- Update of the Conceptual Site Model (CSM) with analytical results collected under the previous SAP (TetraTech 2010)
- Addition of arsenic to the sampling program because of a reduction in the maximum contaminant level from 50 to 10 micrograms per liter (µg/L)
- Addition of natural attenuation parameters to the sampling program
- Update to the sample design to include the addition of two monitoring wells installed under the previous SAP (Tetra Tech 2010)
- Deletion of monitoring well installation requirements in Worksheet 11 of the previous SAP (TetraTech 2010)
- Installation of a new upgradient monitoring well to monitor background concentrations.

The purpose of the SAP is to monitor the nature and extent of volatile organic compounds (VOCs) and metals contaminants exceeding Record of Decision criteria until concentrations are reduced to below the Performance Criteria. The monitoring of 17 groundwater monitoring wells, eight piezometers/staff gauges, and three surface water sampling locations is included in the subject LTMP to achieve this purpose. The groundwater samples will be analyzed for select VOCs and select metals and the surface water samples will be analyzed for total iron, which are the

primary contaminants of concern for these two environmental media at the site. Additionally groundwater samples collected from the monitoring wells will be analyzed for geochemical parameters indicative of natural attenuation. The data will be validated and analyzed for trends consistent with project data quality objectives. Results of the semi-annual sampling events will be summarized in annual groundwater monitoring reports.

Table of Contents

SAP WORKSHEET #1: TITLE AND APPROVAL PAGE	WS 1-1
SAP WORKSHEET #2: SAMPLING AND ANALYSIS PLAN IDENTIFYING INFORMATION	WS 2-1
SAP WORKSHEET #3: DISTRIBUTION LIST	WS 3-1
SAP WORKSHEET #4: PROJECT PERSONNEL SIGN-OFF SHEET	WS 4-1
SAP WORKSHEET #5: PROJECT ORGANIZATIONAL CHART	WS 5-1
SAP WORKSHEET #6: COMMUNICATION PATHWAYS	WS 6-1
SAP WORKSHEET #7: PERSONNEL RESPONSIBILITIES TABLE	WS 7-1
SAP WORKSHEET #8: SPECIAL PERSONNEL TRAINING REQUIREMENTS TABLE	WS 8-1
SAP WORKSHEET #9: PROJECT SCOPING SESSION PARTICIPANTS SHEET	WS 9-1
SAP WORKSHEET #10: CONCEPTUAL SITE MODEL	WS 10-1
SAP WORKSHEET #11: PROJECT QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS STATEMENTS	WS 11-1
SAP WORKSHEET #12: FIELD QUALITY CONTROL SAMPLES	WS 12-1
SAP WORKSHEET #13: SECONDARY DATA CRITERIA AND LIMITATIONS TABLE	WS 13-1
SAP WORKSHEET #14: SUMMARY OF PROJECT TASKS	WS 14-1
SAP WORKSHEET #15: REFERENCE LIMITS AND EVALUATION TABLES	WS 15-1
SAP WORKSHEET #16: PROJECT SCHEDULE/TIMELINE TABLE	WS 16-1
SAP WORKSHEET #17: SAMPLING DESIGN AND RATIONALE	WS 17-1
SAP WORKSHEET #18: LOCATION-SPECIFIC SAMPLING METHODS/SOP REQUIREMENTS TABLE	WS 18-1
SAP WORKSHEET #19: FIELD SAMPLING REQUIREMENTS TABLE	WS 19-1
SAP WORKSHEET #20: FIELD QUALITY CONTROL SAMPLE SUMMARY TABLE	WS 20-1
SAP WORKSHEET #21: PROJECT SAMPLING SOP REFERENCES TABLE	WS 21-1
SAP WORKSHEET #22: FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION TABLE	WS 22-1

SAP WORKSHEET #23: ANALYTICAL SOP REFERENCES TABLE.....	WS 23-1
SAP WORKSHEET #24: ANALYTICAL INSTRUMENT CALIBRATION TABLE.....	WS 24-1
SAP WORKSHEET #25: ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION TABLE	WS 25-1
SAP WORKSHEET #26: SAMPLE HANDLING SYSTEM	WS 26-1
SAP WORKSHEET #27: SAMPLE CUSTODY REQUIREMENTS	WS 27-1
SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE	WS 28-1
SAP WORKSHEET #29: PROJECT DOCUMENTS AND RECORDS TABLE	WS 29-1
SAP WORKSHEET #30: ANALYTICAL SERVICES TABLE	WS 30-1
SAP WORKSHEET #31: PLANNED PROJECT ASSESSMENTS TABLE.....	WS 31-1
SAP WORKSHEET #32: ASSESSMENT FINDINGS AND CORRECTIVE ACTION RESPONSES TABLE.....	WS 32-1
SAP WORKSHEET #33: QUALITY ASSURANCE MANAGEMENT REPORTS TABLE	WS 33-1
SAP WORKSHEETS #34-36: DATA VERIFICATION AND VALIDATION (STEPS I AND IIA/IIB) PROCESS TABLE	WS 34 — 36-1
SAP WORKSHEET #37: USABILITY ASSESSMENT	WS 37-1
REFERENCES	REF-1

Figures

Figure 10-1	Site Layout.....	WS 10-2
Figure 10-2	Concentrations of Iron in Piezometers and Surface Water Locations.....	WS 10-7
Figure 10-3	COCs in Groundwater.....	WS 10-8
Figure 10-4	Geological Cross Section of the Surficial Aquifer	WS 10-19
Figure 10-5	Proposed Groundwater Sampling Location Map	WS 10-22
Figure 10-6	Natural Attenuation Conceptual Site Model	WS 10-24
Figure 11-1	Decision Diagram for Use with Decision Rules	WS 11-8

Tables

Table 10-1	ROD-Specified Groundwater COCS and Corresponding Performance Criteria .	WS 10-3
Table 10-2	Detected Concentrations of Iron in Piezometers	WS 10-9
Table 10-3	Iron Detected in Surface Water.....	WS 10-10
Table 10-4	Detected Concentrations of COCs in Groundwater	WS 10-11
Table 37-1	Data Usability Process.....	WS 37-4

Appendices

Appendix A	DQO Meeting Minutes
Appendix B	Field Standard Operating Procedures
Appendix C	Laboratory Accreditation Certificates
Appendix D	Laboratory Standard Operating Procedures
Appendix E	Laboratory Control Charts

Acronyms and Abbreviations

µg/L	microgram per liter
bgs	below ground surface
°C	Degrees Celsius
CCV	Continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CLEAN	Comprehensive Long-term Environmental Action Navy
COC	Contaminant of concern
CSM	Conceptual site model
CTO	Contract Task Order
CTL	Cleanup Target Level
CV	Calibration verification
DoD	Department of Defense
DoD ELAP	DoD Environmental Laboratory Accreditation Program
DoD QSM	DoD Quality Systems Manual
DO	Dissolved oxygen
DQO	Data quality objective
DVA	Data validation assistant
EB	Equipment blank
EDD	Electronic data deliverable
EICP	Extracted ion current profile
ELAP	Environmental Laboratory Accreditation Program
Empirical	Empirical Laboratories, LLC
eQAPP	Electronic quality assurance project plan
F.A.C.	Florida Administrative Code
FDEP	Florida Department of Environmental Protection
FDOH	Florida Department of Health
FRC	Federal Records Center
FTL	Field team leader
GC	Gas chromatograph
GC/FID	Gas chromatograph/flame ionization detector
GC/MS	Gas chromatograph/mass spectrometer
GCTL	Groundwater Cleanup Target Level
G&M	Geraghty & Miller
GW POC	Groundwater Point of Compliance
HSM	Health and safety manager

Acronyms and Abbreviations (continued)

IAS	Initial Assessment Study
ICAL	Initial calibration
ICP	Inductively coupled plasma
ICV	Initial calibration verification
ID	Identification
IDW	Investigation derived waste
IWTP	Industrial wastewater treatment plant
L	Liter
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
LOD	Limit of detection
LOQ	Limit of quantitation
LTM	Long-term monitoring
LUC	Land-use control
MCL	Maximum contaminant level
µg/L	Microgram per liter
mg/L	Milligram per liter
mL	Milliliter
mV	Millivolt
MPC	Measurement performance criteria
MS	Mass spectrometry
MS/MSD	Matrix spike/matrix spike duplicate
nM	Nano Mole
NA	Not applicable
NAS	Naval Air Station
NAVFAC	Naval Facilities Engineering Command
NCP	National Oil and Hazardous Pollution Contingency Plan
NELAP	National Environmental Laboratory Accreditation Program
NIRIS	Naval Installation Restoration Information Solution
NTU	Nephelometric Turbidity Unit
NWFWMD	Northwest Florida Water Management District
ORP	Oxidation-reduction potential
OU	Operable unit
%D	Percent difference
%R	Percent recovery
%RSD	Percent relative standard deviation
PAH	Polycyclic Aromatic Hydrocarbon
PAL	Project Action Level
PCB	Polychlorinated Biphenyl

Acronyms and Abbreviations (continued)

PM	Project manager
POC	Point of Compliance
PPE	Personal protective equipment
PQO	Project quality objective
PWC	Public Works Center
QA	Quality assurance
QAO	Quality assurance officer
QC	Quality control
QL	Quantitation limit
RCRA	Resource Conservation and Recovery Act
RD	Remedial design
RF	Response factor
RI	Remedial Investigation
ROD	Record of decision
RPD	Relative percent difference
RPM	Remedial project manager
RRT	Relative retention time
SAP	Sampling and analysis plan
SEGS	Southeastern Geological Society
SOP	Standard operating procedure
SSO	Site safety officer
SVOC	Semivolatile organic compound
SWCTL	Surface water cleanup target level
SW POC	Surface water point of compliance
TB	Trip blank
TBD	To be determined
TCL VOC	Target compound list volatile organic compound
TOC	Total organic carbon
TOM	Task order manager
TRPH	Total recoverable petroleum hydrocarbon
TtNUS	Tetra Tech NUS, Inc.
UFP-SAP	Uniform Federal Policy Sampling and Analysis Plan
UFP-QAPP	Uniform Federal Policy for Quality Assurance Plan
U.S. EPA	U.S. Environmental Protection Agency
VOC	Volatile organic compound



SAP WORKSHEET #2: SAMPLING AND ANALYSIS PLAN IDENTIFYING INFORMATION

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: Site 1 Sanitary Landfill

Operable Unit: Operable Unit 1 (OU 1)

Contractor Name: Resolution Consultants

Contract Number: N62470-11-D-8013

Contract Title: Comprehensive Long-term Environmental Action Navy (CLEAN)

Work Assignment No: Contract Task Order (CTO) JM40

1. This sampling and analysis plan (SAP) was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)* (U.S. EPA 2005) and U.S. Environmental Protection Agency (U.S. EPA) *Guidance for Quality Assurance Project Plans, EPA QA/G-5* (U.S. EPA 2002).
2. Identify regulatory program: National Oil and Hazardous Substances Pollution Contingency Plan (NCP); Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA); and Resource Conservation and Recovery Act (RCRA).
3. This SAP is a project-specific SAP.
4. List organizational partners (stakeholders) and identify the connection with lead organization:

Organization Partners/Stakeholders	Connection
U.S. Environmental Protection Agency	Regulatory Oversight
Florida Department of Environmental Protection (FDEP)	Regulatory Oversight
Naval Facilities Engineering Command (NAVFAC) Southeast	Operator/Lead Agency
Naval Air Station (NAS) Pensacola	Property Owner, Navy Contractor



5. Lead organization: NAVFAC Southeast U.S. Department of Navy

6. If any required SAP elements and required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below: Not Applicable — All worksheets are applicable.



SAP WORKSHEET #3: DISTRIBUTION LIST

(UFP-QAPP Manual Section 2.3.1)

Sampling and Analysis Plan Recipients	Title	Organization	Telephone Number	E-mail Address
Patty Marajh-Whittemore	Remedial Project Manager (RPM)	NAVFAC Southeast NAS Jacksonville Building 903 Jacksonville, Florida 32212	904-542-6202	patty.whittemore@navy.mil
Ken Bowers	Quality Assurance Officer/Chemist	NAVFAC Atlantic 6505 Hampton Boulevard Norfolk, Virginia 23508	757-322-8341	kenneth.a.bowers@navy.mil
Greg Campbell	Remedial Project Manager (RPM)	NAS Pensacola 310 John Tower Road Pensacola, Florida 32508-5000	850-452-3131 Ext. 3007	gregory.campbell@navy.mil
Tim Woolheater	Senior Remedial Project Manager (RPM)	U.S. EPA Atlanta Federal Center 61 Forsyth Street, SW Atlanta, Georgia 30303-8960	404-562-8510	woolheater.tim@epamail.epa.gov
David Grabka	Remedial Project Manager (RPM)	FDEP MS 4535 2600 Blair Stone Road Tallahassee, Florida 32399-2400	850-245-8997	david.grabka@dep.state.fl.us
Mike Singletary	Environmental Technical Support	NAVFAC SE NAS Jacksonville Building 903 Jacksonville, Florida 32212	904-542-6303	michael.a.singletary@navy.mil
Librarian	Head of Reference Desk (NAS Pensacola Administrative Record)	John C. Pace Library University of West Florida	850-474-2424	Librarian John C. Pace Library University of West Florida 11000 University Parkway Pensacola, Florida 32514
Tina Cantwell	Project Chemist/Quality Assurance Officer (QAO)/Data Manger	Resolution Consultants 5724 Summer Trees Drive Memphis, Tennessee	901-937-4315	tcantwell@ensafe.com
Bill Honea	Field Team Leader (FTL)/Site Safety Officer (SSO)	Resolution Consultants 308 North Peters Road, Suite 200 Knoxville, Tennessee 37922	865-693-3623	whonea@ensafe.com



Sampling and Analysis Plan Recipients	Title	Organization	Telephone Number	E-mail Address
Bill Duffy	Sampling Specialist	Resolution Consultants 4300 Legendary Drive Building C, Suite 240 Destin , Florida 32541	850-269-2200	bill.duffy@aecom.com
Allison Harris	Task Order Manager (TOM)	Resolution Consultants 5724 Summer Trees Drive Memphis, Tennessee 38134	901-372-7962	aharris@ensafe.com
John Knopf	Health & Safety Manager (HSM)	Resolution Consultants 5724 Summer Trees Drive Memphis, Tennessee 38134	901-372-7962	jknopf@ensafe.com
Gerry Walker	Project Manager/Technical Advisor	Tetra Tech1558 Village Square Boulevard, Suite 2 Tallahassee, Florida 32308	850-385-9866 Ext. 1362	gerry.walker@tetrattech.com
Sam Naik	Project Manager/Technical Advisor	CH2M Hill 1000 Abernathy Road Suite 1600 Atlanta, Georgia 30368	678-530-4248	sam.naik@ch2m.com
Sonya Gordon	Laboratory Project Manager	Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270 Nashville, Tennessee 37228	615-345-1115	sgordon@empirlabs.com
Robbin Robl	Laboratory Project Manager	Pace Analytical Energy Services, LLC (Microseeps, Inc.) 220 William Pitt Way Pittsburgh, Pennsylvania 15238	412-826-5245	rrobl@microseeps.com

Notes:

FDEP = Florida Department of Environmental Protection
 FTL = Field Team Leader
 NAVFAC = Department of the Navy, Naval Facilities Engineering Command, Southeast
 NAS = Naval Air Station
 QAO = Quality Assurance Officer
 RMP = Remedial Project Manager
 TOM = Task Order Manager
 U.S. EPA = U.S. Environmental Protection Agency
 HSM = Health & Safety Manager



SAP WORKSHEET #4: PROJECT PERSONNEL SIGN-OFF SHEET

(UFP-QAPP Manual Section 2.3.2)

Certification that project personnel have read the text will be obtained by the following methods:

E-mails will be sent to Navy, Resolution Consultants, regulatory agencies, and subcontractor project personnel, who will be requested to verify by e-mail that they have read the applicable SAP/sections and the date on which they were reviewed. Copies of the verification e-mail will be included in the project files and identified in Worksheet #29. Copies of regulatory agency comments, if received, will be retained in the project files and are listed in Worksheet #29 as project records.

A copy of the signed Worksheet #4 will be retained in the project files and identified as a project document in Worksheet #29.



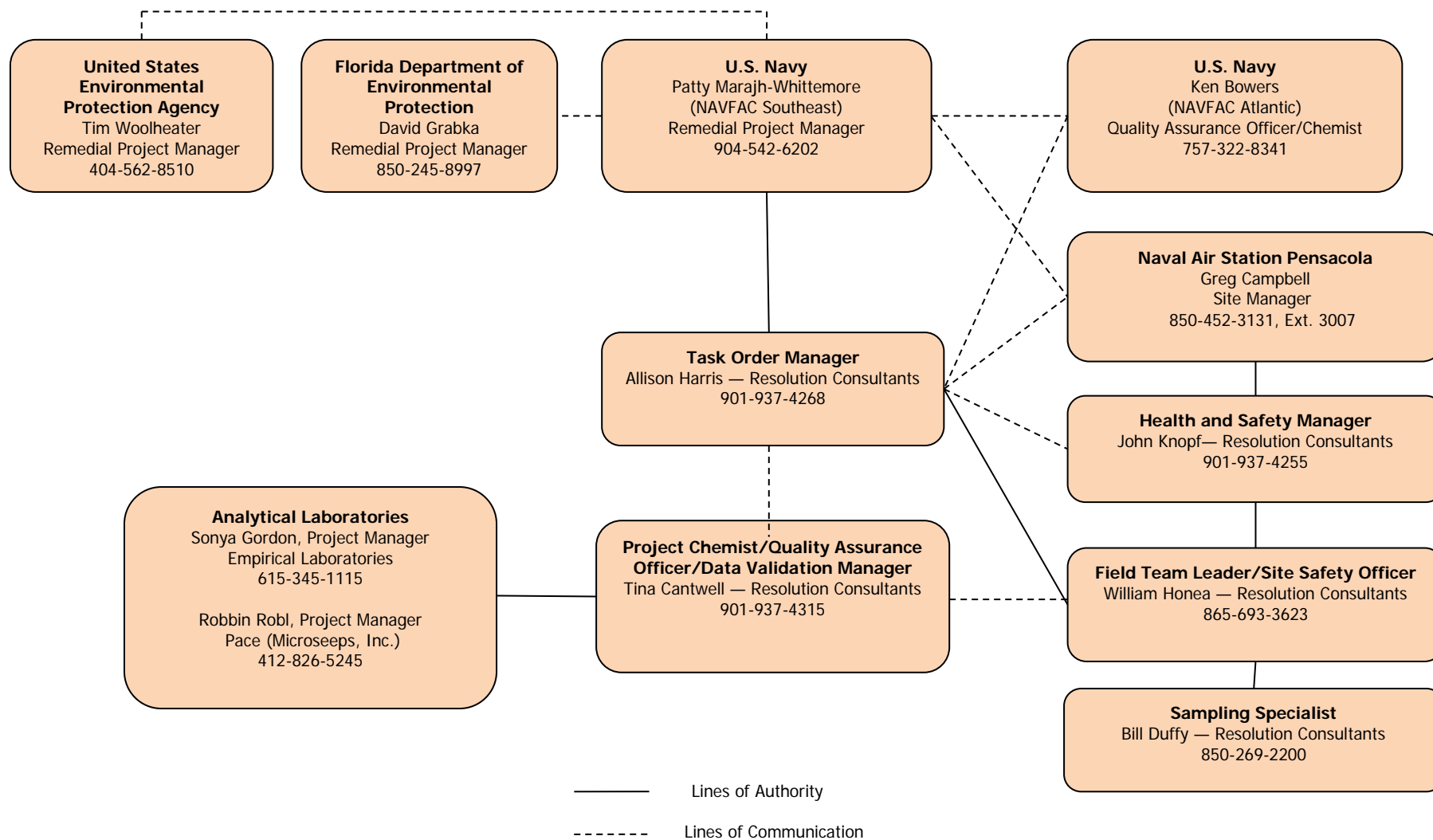
Project Personnel Sign-Off Sheet					
Name	Organization/Title/Role	Telephone Number	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Navy and Regulator Project Team Personnel					
Patty Marajh-Whitemore	NAVFAC Southeast/Remedial Project Manager	904-542-6202		All	
Greg Campbell	NAS Pensacola Site Manager/Point of Contact	850-452-3131 Ext. 3007		All	
Tim Woolheater	U.S. EPA/Remedial Project Manager	404-562-8510		All	
David Grabka	FDEP/Remedial Project Manager	850-245-8997		All	
Resolution Consultants Project Team Personnel					
Allison Harris	Resolution Consultants/Task Order Manager	901-937-4268		All	
William Honea	Resolution Consultants/Field Team Leader/ Site Safety Officer	865-693-3623		All	
Bill Duffy	Resolution Consultants Sampling Specialist	850-269-2200		All	
Tina Cantwell	Resolution Consultants/Chemist/Quality Assurance Officer/Data Manager	901-937-4315		All	
Subcontractor Personnel					
Sonya Gordon	Empirical Laboratories Project Manager	615-345-1115		Worksheets #6, #12, #14, #15, #19, #23-28, #30, and #34-36	
Robbin Robl	Pace Analytical Energy Services LLC (Microseeps, Inc.) Project Manager	412-826-5245		Worksheets #6, #14, #15, #19, #23-28, #30, and #34-36	

Notes:

FDEP = Florida Department of Environmental Protection
 NAVFAC = Naval Facilities Engineering Command
 NAS = Naval Air Station
 U.S. EPA = United States Environmental Protection Agency

SAP WORKSHEET #5: PROJECT ORGANIZATIONAL CHART

(UFP-QAPP Manual Section 2.4.1)





SAP WORKSHEET #6: COMMUNICATION PATHWAYS

(UFP-QAPP Manual Section 2.4.2)

The communication pathways for the SAP are shown below. Communications described in this worksheet will be documented in the appropriate format for the specific interaction. Appropriate forms of documentation include, but are not limited to: email, telephone logs, field logbook notations, field forms, audit reports, and formal memoranda, or as described below. Communications will be part of the project records and will be maintained in the locations described in Worksheet #29.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Regulatory Agency Interface	Navy RPM	Patty Marajh-Whittemore	904-542-6202	The Navy RPM informs regulatory agency of work progress on a periodic basis.
Progress Reports	Resolution Consultants FTL/SSO Resolution Consultants TOM Navy RPM	William Honea Allison Harris Patty Marajh-Whittemore	865-693-3623 901-937-4268 904-542-6202	FTL verbally informs the TOM on a daily basis field updates. TOM provides a weekly update to the RPM either by phone message and/or e-mail each Friday afternoon field activities are taking place.
Gaining Site Access	Resolution Consultants FTL NAS Pensacola Site Manager	William Honea Greg Campbell	865-693-3623 850-452-3131, Ext. 3007	The Resolution Consultants FTL will contact the NAS Pensacola point of contact verbally or via e-mail at least 3 days prior to commencement of field work to arrange for access to the site for all field personnel.
Stop Work due to Safety Issues	Resolution Consultants FTL/SSO Resolution Consultants TOM Resolution Consultants HSM Navy RPM NAS Pensacola Site Manager	William Honea Allison Harris John Knopf Patty Marajh-Whittemore Greg Campbell	865-693-3623 901-937-4268 901-937-4255 904-542-6202 850-452-3131 Ext. 3007	Any field team member who observes an unsafe situation has the authority to stop work. If Resolution Consultants is the responsible party for a stop work command, the Resolution Consultants SSO will inform onsite personnel, subcontractor(s), the NAS Pensacola site manager, and the identified Partnering Team members within 1 hour (verbally or by e-mail). If a subcontractor is the responsible party, the subcontractor PM must verbally inform the Resolution Consultants SSO within 15 minutes, and the Resolution Consultants SSO will then follow the procedure listed above.
SAP Changes prior to Field/Laboratory work	Resolution Consultants TOM Navy RPM U.S. EPA RPM FDEP RPM	Allison Harris Patty Marajh-Whittemore Tim Woolheater David Grabka	901-937-4268 904-542-6202 404-562-8510 850-245-8997	Any change of the approved SAP will be made only upon authorization of the Navy RPM and regulatory agencies. The Resolution Consultants TOM is responsible for initiating any SAP change requests via the communication channels described for the Navy and regulatory agencies.



Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
SAP Changes in the Field	Resolution Consultants FTL/SSO Resolution Consultants TOM Navy RPM U.S. EPA RPM FDEP RPM	William Honea Allison Harris Patty Marajh-Whittemore Tim Woolheater David Grabka	865-693-3623 901-937-4268 904-542-6202 404-562-8510 850-245-8997	FTL informs TOM verbally within same day; TOM informs Navy RPM via e-mail within 24 hours; TOM sends a concurrence letter to RPM, if warranted, within 7 calendar days and RPM signs the letter within 5 business days of receipt. Scope change is to be implemented before work is executed. Document the change on a field task modification request form (within 2 business days) or SAP amendment (within timeframe agreed to by Project Team). Any change of the approved SAP, effecting the scope or implementation of the sampling program, will be made only upon authorization of the Navy RPM and regulatory agencies.
Field Corrective Actions	Resolution Consultants FTL/SSO Resolution Consultants TOM Navy RPM U.S. EPA RPM FDEP RPM	William Honea Allison Harris Patty Marajh-Whittemore Tim Woolheater David Grabka	865-693-3623 901-937-4268 904-542-6202 404-562-8510 850-245-8997	FTL informs TOM verbally within same day; TOM informs Navy RPM via e-mail within 24 hours that corrective actions have been implemented. Corrective actions will be documented in weekly progress reports. Significant corrective actions will be communicated to the regulatory agencies.
Recommendations to stop work and initiate work upon corrective action	Resolution Consultants FTL/SSO Resolution Consultants TOM Resolution Consultants QAO Navy RPM U.S. EPA RPM FDEP RPM	William Honea Allison Harris Tina Cantwell Patty Marajh-Whittemore Tim Woolheater David Grabka	865-693-3623 901-937-4268 901-937-4315 904-542-6202 404-562-8510 850-245-8997	Responsible party verbally informs the TOM, FTL, and subcontractors within 1 hour of recommendation to stop work and within 24 hours of recommendation to restart work. Responsible party follows verbal notification with an e-mail to the Project Team within 24 hours. Significant corrective actions will be communicated to the regulatory agencies.
Sample Receipt and Laboratory Quality Variances	Empirical Laboratories PM Pace (Microseeps, Inc.) PM Resolution Consultants FTL Resolution Consultants TOM	Sonya Gordon Robbin Robl Brian Caldwell Allison Harris	615-345-1115 412-826-5245 865-693-3623 901-937-4268	<p>The Laboratory PM will notify (verbally or via e-mail) the Resolution Consultants FTL immediately upon receipt of any chain of custody/sample receipt variances for clarification or direction from the Resolution Consultants FTL.</p> <p>The Resolution Consultants FTL will notify (verbally or via e-mail) the Resolution Consultants TOM within 1 business day, if corrective action is required.</p> <p>The Resolution Consultants TOM will notify (verbally or via e-mail) the Laboratory PM and the Resolution Consultants FTL within 1 business day of any required corrective action.</p>
Analytical Corrective Actions	Empirical Laboratories PM Pace (Microseeps, Inc.) PM Resolution Consultants Project Chemist	Sonya Gordon Robbin Robl Tina Cantwell	615-345-1115 412-826-5245 901-937-4315	The laboratory shall notify the Resolution Consultants' chemist of any analytical data anomaly within 1 business day of discovery. After the laboratory receives guidance from Resolution Consultants' chemist, the laboratory shall initiate any corrective action to prevent further anomalies.



Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Analytical Data Quality Issues	Empirical Laboratories PM Pace (Microseeps, Inc.) PM Resolution Consultants Project Chemist Resolution Consultants TOM Navy RPM	Sonya Gordon Robbin Robl Tina Cantwell Allison Harris Patty Marajh-Whitemore	615-345-1115 412-826-5245 901-937-4315 901-937-4268 904-542-6202	<p>The laboratory PM notifies (verbally or via e-mail) the Resolution Consultants' chemist within 1 business day of when an issue related to laboratory data is discovered. Resolution Consultants' chemist notifies Resolution Consultants' TOM within 1 business day.</p> <p>Resolution Consultants' chemist notifies the Resolution Consultants' TOM verbally or via e-mail within 48 hours of validation completion that a non-routine and significant laboratory quality deficiency has been detected that could affect this project and/or other projects. Resolution Consultants' TOM verbally advises the Navy RPM within 24 hours of notification from the chemist. The Navy RPM takes corrective action that is appropriate for the identified deficiency.</p>
Reporting Data Validation Issues/Data Validation Corrective Actions	Resolution Consultants Project Chemist Resolution Consultants TOM	Tina Cantwell Allison Harris	901-937-4315 901-937-4268	<p>The Resolution Consultants project chemist or data validator will perform validation as specified in Worksheets #34, #35, and #36, and will contact the laboratory as soon as possible if issues are found that require corrective action.</p> <p>If the Resolution Consultants project chemist or data validator identifies non-usable data during the data validation process that requires corrective action, the Resolution Consultants TOM will coordinate with the project chemist to take corrective action appropriate for the identified deficiency to ensure the project objectives are met. Corrective action may include resampling and/or reanalyzing the affected samples, as determined by the Resolution Consultants TOM.</p>



Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Notification of Non-Usable Data	Empirical Laboratories PM Pace (Microseeps Inc.) PM Resolution Consultants Project Chemist Resolution Consultants TOM NAVFAC SE RPM U.S. EPA RPM FDEP RPM	Sonya Gordon Robbin Robl Tina Cantwell Allison Harris Patty Marajh-Whitemore Tim Woolheater David Grabka	615-345-1115 412-826-5245 901-937-4315 901-937-4268 904-542-6202 404-562-8510 850-245-8997	<p>If the laboratory determines that any data they have generated is non-usable, the Laboratory PM will notify (verbally or via e-mail) the Resolution Consultants project chemist within 1 business day of when the issue is discovered.</p> <p>The Resolution Consultants project chemist will notify (verbally or via e-mail) Resolution Consultants TOM within 1 business day of the need for corrective action, if the non-usable data is a significant issue (i.e., critical sample data).</p> <p>Corrective action may include resampling and/or reanalyzing the effected samples.</p> <p>If a Resolution Consultants project chemist or data validator identifies non-usable data during the data validation process, the TOM will be notified verbally or via e-mail within 48 hours of validation completion that a non-routine and significant laboratory quality deficiency has resulted in non-usable data.</p> <p>The Resolution Consultants TOM will take corrective action appropriate for the identified deficiency to ensure the project objectives are met. The Resolution Consultants TOM will notify (verbally or via email) the NAVFAC RPM on any problems with the laboratory or analysis that could significantly affect the usability of the data or project failures that impact the ability to complete the scope of work. The NAVFAC RPM, may at their discretion, contact the Navy project chemist for assistance in problem resolution. Such notification will be made within 1 business day of when the issue is discovered. The NAVFAC RPM will notify the U.S. EPA and FDEP when any significant corrective action is taken within 3 days.</p>

Notes:

RPM	=	Remedial project manager	SSO	=	Site safety officer
FTL	=	Field team leader	HSM	=	Health and safety manager
TOM	=	Task order manager	SAP	=	Sampling and analysis plan
QAO	=	Quality assurance officer	PM	=	Project manager
U.S. EPA	=	United States Environmental Protection Agency	FDEP	=	Florida Department of Environmental Protection
NAVFAC	=	Naval Facilities Engineering Command	NAS	=	Naval Air Station



SAP WORKSHEET #7: PERSONNEL RESPONSIBILITIES TABLE

(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities
Patty Marajh-Whittemore	Remedial Project Manager	NAVFAC Southeast	Oversees project implementation, including scoping, data review, and evaluation.
Ken Bowers	Quality Assurance Officer/Chemist	NAVFAC Atlantic	Provides quality control and chemistry oversight.
Greg Campbell	NAS Pensacola Site Manager	NAS Pensacola Public Works Center	Oversees Base activities, including field work coordination, scoping, data review, and evaluation.
Tim Woolheater	Remedial Project Manager	U.S. EPA	Participates in scoping, data review, evaluation, and review and approval of the SAP. Oversees project execution for U.S. EPA.
David Grabka	Remedial Project Manager	FDEP	Participates in scoping, data review, evaluation, and review and approval of the SAP. Oversees project execution for FDEP.
Allison Harris	Task Order Manager	Resolution Consultants	Oversees project, financial, schedule, and technical day to day management of the project.
William Honea	Field Team Leader/ Site Safety Officer	Resolution Consultants	As the FTL, supervises, coordinates, and performs and documents field sampling activities. As the SSO, is responsible for onsite project-specific health and safety training and monitoring site conditions.
Bill Duffy	Sampling Specialist	Resolution Consultants	Performs and documents sampling activities, packs samples, prepared chain-of-custody records.
John Knopf	Health and Safety Manager	Resolution Consultants	Responsible for providing health and safety training for all personnel and approving the site-specific Health and Safety Plan. Prepares health and safety reports for management.
Tina Cantwell	Project Chemist/QAO/Data Validation Manager	Resolution Consultants	As project chemist, prepares laboratory scopes of work, and coordinates laboratory related functions with laboratory. Performs or oversees data quality reviews and quality assurance of data validation deliverables. As QAO, ensures quality aspects of the project are implemented, documented, and maintained. As data validation manager, performs or oversees data validation and data input in both the project database and the Navy's Naval Installation Restoration Information Solution database.
Sonya Gordon	Project Manager/ Analytical Laboratory	Empirical Laboratories, LLC.	Coordinates analyses with laboratory staff, ensures that scope of work is followed, provides quality review of data packages, and communicates with Resolution Consultant project staff.
Robbin Robl	Project Manager/ Analytical Laboratory	Pace (Microseeps, Inc.)	Coordinates analyses with laboratory staff, ensures that scope of work is followed, provides quality review of data packages, and communicates with Resolution Consultant project staff.

Notes:

NAVFAC = Naval Facilities Engineering Command
 U.S. EPA = United States Environmental Protection Agency
 SAP = Sampling and analysis plan
 SSO = Site safety officer
 NAS = Naval Air Station

FDEP = Florida Department of Environmental Protection
 FTL = Field team leader
 QAO = Quality assurance officer



SAP WORKSHEET #8: SPECIAL PERSONNEL TRAINING REQUIREMENTS TABLE

(UFP-QAPP Manual Section 2.4.4)

All field personnel will have appropriate training to conduct the field activities to which they are assigned. Additionally, each site worker will be required to have completed appropriate Hazardous Waste Operations and Emergency Response training specified in Occupational Safety and Health Administration 29 Code of Federal Regulations 1910.120(e).



SAP WORKSHEET #9: PROJECT SCOPING SESSION PARTICIPANTS SHEET

(UFP-QAPP Manual Section 2.5.1)

Project/Site Name: NAS Pensacola, OU 1 — Site 1 Sanitary Landfill					
Site Location: Teleconference					
Date of Session: May 14, 2009					
Scoping Session Purpose: Develop Data Quality Objectives to support UFP-QAPP development					
Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Patty Marajh-Whittemore	NAVFAC SE RPM	NAVFAC SE	904-542-6964	patty.whittemore@navy.mil	Navy RPM
Greg Campbell	NAS Pensacola RPM	NAS Pensacola	850-452-3131 Ext. 3007	gregory.campbell@navy.mil	NAS Pensacola RPM
Tracie Bolaños	Federal Facilities RPM	FDEP	850-245-8998	tracie.bolanos@dep.stat.fl.us	FDEP RPM
Greg Fraley	RPM	U.S. EPA	404-562-8544	fraley.gregory@epa.gov	U.S. EPA RPM
Jon Tucker	Government Chemist	NAVFAC	757-322-8288	jonathan.tucker@navy.mil	Navy Quality Assurance
Mike Singletary	Senior Engineer	NAVFAC SE	904-542-6303	michael.a.singletary@navy.mil	Technical Support
Gerry Walker	Task Order Manager	TtNUS	850-385-9866 Ext. 1362	gerry.walker@tetrattech.com	Project Management
Katie Newman	Technical Support Staff	TtNUS	850-385-9866 Ext. 1357	katie.newman@tetrattech.com	Technical Support
Tom Johnston	Quality Assurance Manager	TtNUS	412-921-8615	tom.johnston@tetrattech.com	Data Quality Objective Facilitator

Notes:

UFP-QAPP = Uniform Federal Policy for Quality Assurance Project Plan
 NAVFAC = Naval Facilities Engineering Command
 SE = Southeast
 RPM = Remedial Project Manager
 NAS = Naval Air Station
 FDEP = Florida Department of Environmental Protection
 U.S. EPA = U.S. Environmental Protection Agency
 TtNUS = Tetra Tech NUS, Inc.

Comments/Decisions: The Project Team decided that a follow-up meeting would be required to address questions concerning the potential for the eventual discontinuation of monitoring for this site, which has waste in place and no engineered cap. DQO Meeting Minutes are provided in Appendix A.



Action Items:

- Tom Johnston will send presumptive remedy guidance for landfills to the Project Team.
- Tracie Bolaños will review the "Technical Memorandum, Reconnaissance Phase Flow Control Pilot Study for OU1", (TtNUS 2009) (referred to in this SAP as the "Tech Memo"), concerning a proposed surface water threshold value and possibly refer the Tech Memo to the University of Florida for evaluation and concurrence on the proposed threshold value.

Consensus Decisions:

- New work must implement the Record of Decision (ROD), which is a legal document and is binding.
- Wetland 3 (immediately east of the landfill) is essentially an extension of the landfill and therefore does not have to be protected as would a normal wetland. Protection of Wetland 4D, and especially Bayou Grande since it an estuary of Pensacola Bay and a Class III surface water, is most important. The surface water point of compliance would be upstream of Bayou Grande.



SAP WORKSHEET #10: CONCEPTUAL SITE MODEL

(UFP-QAPP Manual Section 2.5.2)

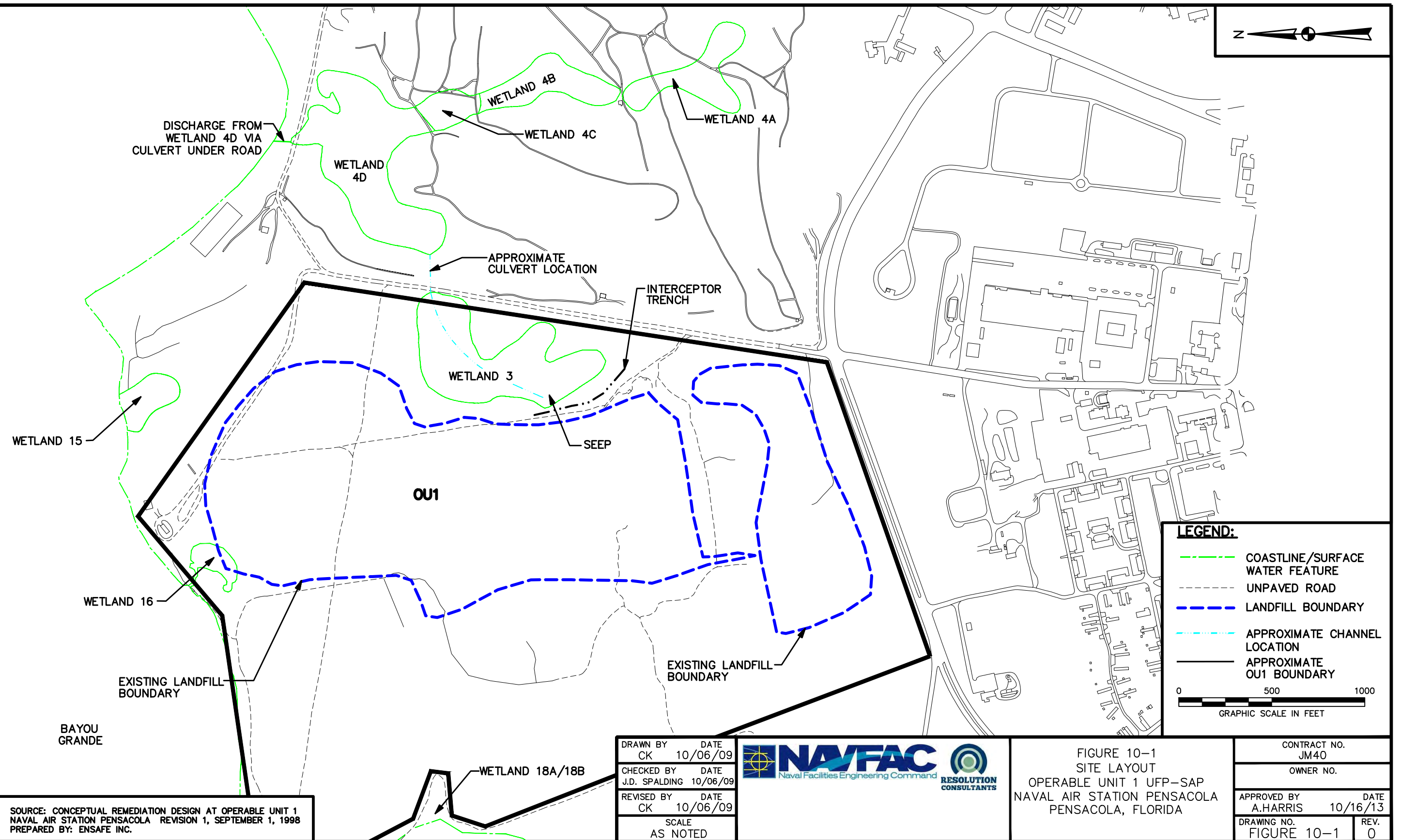
10.1 Background/Site History

The United States Navy has maintained a presence in the Pensacola area since 1825 when a Navy yard was established on Pensacola Bay. Between 1828 and 1835, the Navy acquired approximately 2,300 acres as operations expanded. Several natural disasters in the early 1900s destroyed the yard and forced it into maintenance status in 1911. Three years later, the Navy's first permanent air station was established on the site of the old Navy yard. The air station has been the primary training base for naval aviators since that time and the base continues to expand. The current NAS Pensacola facility consists of 5,800-acres.

OU 1, also referred to as Site 1 — Sanitary Landfill, is a former sanitary landfill located on the NAS Pensacola facility, in northwest Florida on the west edge of Pensacola Bay, two miles south of Pensacola, Escambia County, Florida. The former landfill operated between 1950 and 1976 and encompasses an area of approximately 80 acres that is bordered on the north by an estuary of Pensacola Bay named Bayou Grande (Figure 10-1). The landfill is oblong and oriented north to south. The southern segment of the landfill is the oldest and was used in the 1950s. The larger, northern segment was constructed in the 1960s. The central portion of the landfill operated from the 1960s into the 1970s, until the landfill was officially closed on October 1, 1976.

The land is currently used as a landfill and limited recreational area with designated nature trails. OU 1 elevations range between 8 to 20 feet above mean sea level. At least 2 feet of clean soil covers the entire landfill. An engineered cap has not been installed. The landfill is vegetated with pine trees and scrub brush that serve to stabilize the soil cover. Soils in the landfill, covering the landfill, and in surrounding terrain are relatively permeable and typical of soils found in Florida. The permeable soils generally prevent development of streams at this site because they minimize surface runoff. Multiple wetlands are present near the landfill (see Figure 10-1) including Wetland 15 to the northeast, Wetlands 16 and 17 to the north, Wetland 18A/18B to the west, and Wetlands 3-4A-4B-4C-4D to the east. Environmental impacts to wetlands within NAS Pensacola are collectively addressed through the OU 1, OU 2, and OU 16 Wetland SAP which is currently under development. Wetlands 16 and 17 were screened out during the Site 41 — OU 16 RI/FS process and will not be assessed further. Additionally, Figure 10-1 notes the location of the former interceptor trench which was located between Wetland 3 and the landfill. The use of the interceptor trench has been discontinued due to the inability of the trench to effectively reduce iron impacts to Wetland 3. Wetland 3 is located immediately east of the landfill. Wetland 3 discharges into Wetland 4D, which discharges at its northern edge into Bayou Grande.

ACAD:0702\6-24-08 Groundwater\0702-G01.dwg 10/06/09 CK PIT





Reportedly, the OU 1 landfill contains over 60,000 tons of various industrial and construction wastes such as polychlorinated biphenyls (PCBs) and transformer oil-soaked rags, paint chips, paint sludge and paint booth filter pads, ketone-soaked rags, compressed air cylinders, asbestos, wood soaked with plating solution, pesticide rinses, garbage, wastes from outlying facilities, and containers from paints, mercury, pesticides, oils, strippers, plating chemicals, solvents, and thinners.

Based on past investigations and the 1998 ROD, contaminants of concern (COCs) were established for the site. Table 10-1 lists the COCs that require long-term monitoring:

Table 10-1 ROD-Specified Groundwater COCs and Corresponding Performance Criteria	
Contaminant	Performance Criteria (µg/L)
Arsenic*	10
Nickel	100
Benzene	1
Chlorobenzene	100
Vinyl Chloride	1

Note:

- * Arsenic was not identified in the Record of Decision (ROD), because all detected concentrations of arsenic were below 50 µg/L (Maximum Contaminant Level [MCL]). The MCL subsequently dropped to 10 micrograms per liter (µg/L). Arsenic was detected in groundwater during the remedial investigation at concentrations greater than 10 µg/L; therefore, arsenic will be added as a COC using a ROD Amendment.

Data from several previous investigations are summarized below. The full names of each report referred to in this section are provided in the References section at the end of this document.

1974 to 1983: Leachate was discovered in 1974 to be exiting the landfill into a nearby golf course pond from an abandoned drainage field (Geraghty & Miller [G&M] 1984). An Initial Assessment Study (IAS) completed by the Naval Energy and Environmental Support Activity ([NEESA 1983]) concluded that the landfill posed a potential threat to human health and the environment and warranted further investigation in a verification study.

1984 to 1986: A Verification Study (G&M 1984) detected organic chemicals in all groundwater samples, most of which were volatile organic compounds (VOCs). The highest VOC concentrations were associated with the central portion of OU 1. No PCBs or pesticides were detected, and semivolatile organic compounds (SVOCs) were detected at low concentrations. All detected metals concentrations were less than the applicable 1984 FDEP drinking water standards. A Characterization Study (G&M 1986) verified organic chemical contamination in groundwater, with detectable concentrations of at least one VOC in 12 of 16 wells. SVOCs, PCBs, and pesticides were not detected.



1991: In a Contamination Assessment/Remedial Activities Investigation (Ecology and Environment, Inc. [E&E] 1991), 15 permanent and 28 temporary wells were sampled. Groundwater, surface water, sediment, and soil samples were collected and analyzed for full Target Compound List (TCL) VOCs, TCL SVOCs, TCL pesticides/PCBs, Target Analyte List (TAL) metals, and cyanide; total recoverable petroleum hydrocarbon (TRPH); and gross alpha radioactivity. Sediments contained elevated TRPH, polycyclic aromatic hydrocarbon (PAH), phenol, and metals concentrations, which were attributed to landfilling activities. Select metals and chlorobenzene were detected. Significant VOCs, PAHs, and phenol concentrations were detected in two surface soil samples (representing a relict tar pit and a collapse feature), both of which contained tar-like material. The material, which was tested for toxicity characteristic leachate procedure and deemed non-hazardous, was removed and placed in a Subtitle D landfill. Significant concentrations of one or more of the following chemicals were detected in groundwater: VOCs, phenols, SVOCs, metals, and TRPH. Pesticides, PCBs, and cyanide were not detected in any groundwater samples. Metals and VOCs concentrations exceeded Florida drinking water standards in several shallow well groundwater samples, and three deep well groundwater samples contained low levels of VOCs and SVOCs. The upper portion of the surficial aquifer extends from land surface to a depth of approximately 45 ft; a downward hydraulic gradient across the site indicated a potential for downward contaminant migration within this upper portion of the surficial aquifer.

1992-1996: Results of the Site 1 Final Remedial Investigation (RI) Report (EnSafe/Allen & Hoshall [E/A&H] 1996) identified the following contaminants:

- Wetland Sediments — metals, pesticides, and SVOCs
- Wetland Surface Water — metals and SVOCs
- Bayou Grande Sediments — metals, pesticides, and SVOCs
- Bayou Grande Surface Water — metals only
- Groundwater — metals and VOCs (shallow wells)

1997 to 1999: A Proposed Plan was submitted for public comment; a Final ROD was issued on September 25, 1998 (EnSafe 1998); 73 tons of landfill soil and debris were removed in 1998; and a Final Remedial Design (RD) was issued in 1999 (EnSafe 1999).



The *Long Term Monitoring Plan (LTMP) for Phase II Remedial Action* was issued by Bechtel Environmental Inc. in July 1999. Semi-annual sampling events have been conducted since March 2000, but were not conducted from November 2003 through May 2010. LTM was not conducted from 2004 to 2005 due to extensive hurricane damage throughout the facility. Later, an Optimization Study was conducted (final report submitted April 2008) causing further delays. In addition, the Reconnaissance Phase Flow Control Pilot Study (which was conducted to evaluate the effectiveness and impacts of blocking the culvert that connects Wetland 3 and 4D) was not completed until 2009, adding to additional delays while the study temporarily changed hydraulic conditions at the site. After review and approval of the Data Quality Objectives and Uniform Federal Policy Sampling and Analysis Plan (UFP-SAP), submitted in July 2010 and approved in November 2010, LTM has continued.

As identified in the Optimization Study (Tetra Tech 2008), benzene and vinyl chloride are contaminants that have shown persistent, stable concentrations above the Florida GCTLs from Chapter 62-777, Florida Administrative Code (F.A.C.) in many wells across the site. The extent of both of these contaminants has typically remained stable at the site since sampling was initiated in 2000. The concentrations of benzene and vinyl chloride were present at relatively low levels, ranging from not detected to 69 micrograms per liter ($\mu\text{g/L}$) and not detected to 42 $\mu\text{g/L}$, respectively, over the 10 sampling events that were conducted between March 2000 and December 2005.

Benzene and vinyl chloride have been used as the target or indicator organic contaminants. Benzene has also been detected at these five locations (01GGM33, 01GI41, 01GI59, 01GS62, and 01GI65 (Tetra Tech 2008). Reviewing the historical chemical data, the concentration trends appear to be stable and decreasing at these respective wells.

The geochemical parameters indicate that the conditions in the intermediate (approximately 40 ft deep) wells are mildly or moderately reducing, whereas the geochemistry is generally oxidizing in shallow wells (approximately 25 ft deep) when evaluated at the same well cluster. The presence of oxidizing conditions in the shallow interval likely persists due to direct recharge of oxygenated water via precipitation infiltration. The persistence of volatile contaminants in the intermediate monitored interval is likely due to the slow degradation rates of benzene and vinyl chloride under reducing conditions. The organic contaminant plumes monitored at the site do not appear to be increasing in concentration or expanding in areal extent.

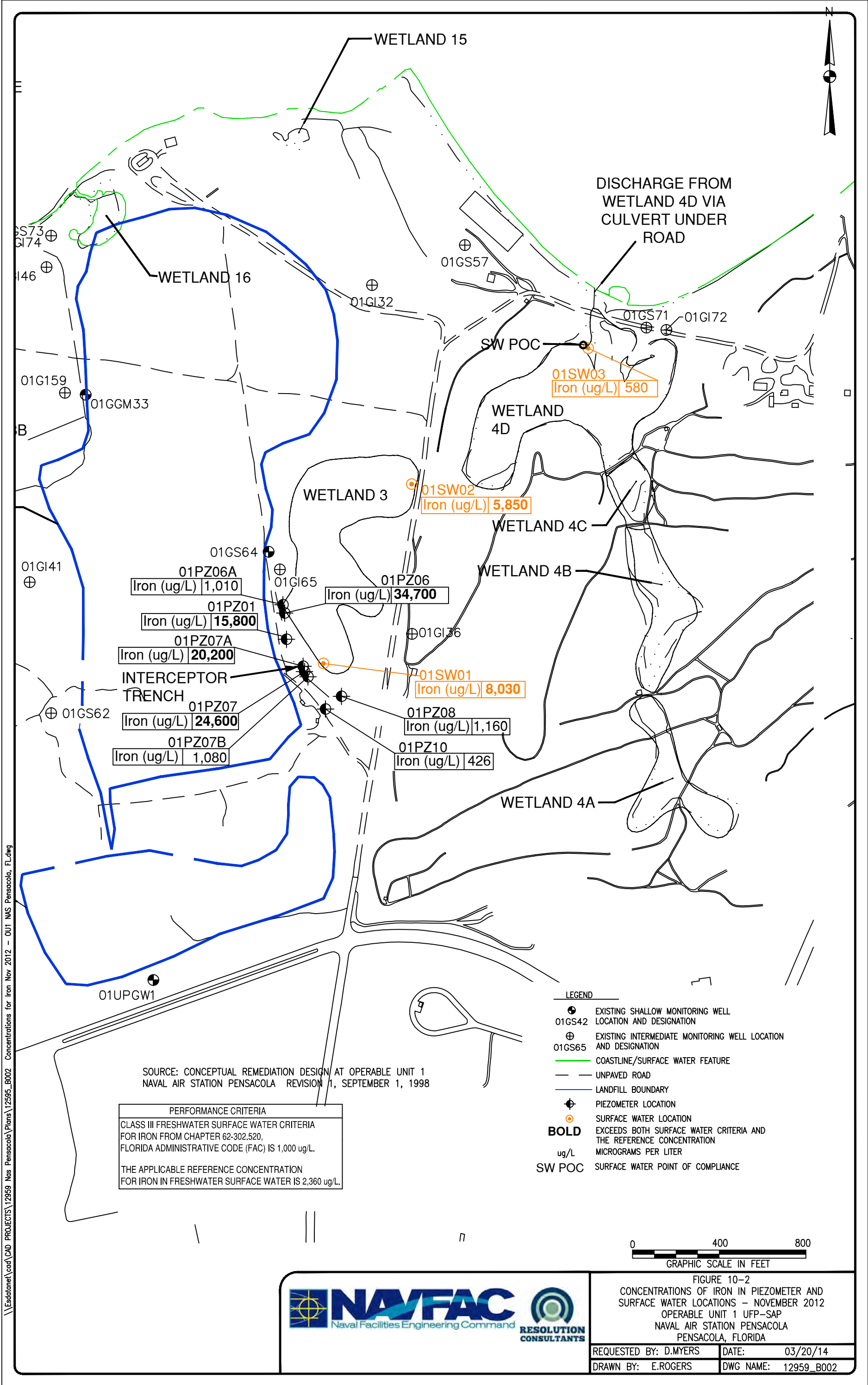


Analytical results for all surface water and groundwater sampling events from August 2000 — January 2012 are presented in Table 10-2 for iron at piezometers, Table 10-3 for iron in surface water, and Table 10-4 for groundwater COCs. The tables are excerpted from the November 2012 Groundwater Sampling Event Data for Operable Unit 1 (Resolution Consultants, 17 April 2013). Figure 10-2 presents piezometer and surface water iron results from the November 2012 sampling event and Figure 10-3 presents groundwater COCs results from the November 2012 sampling event.

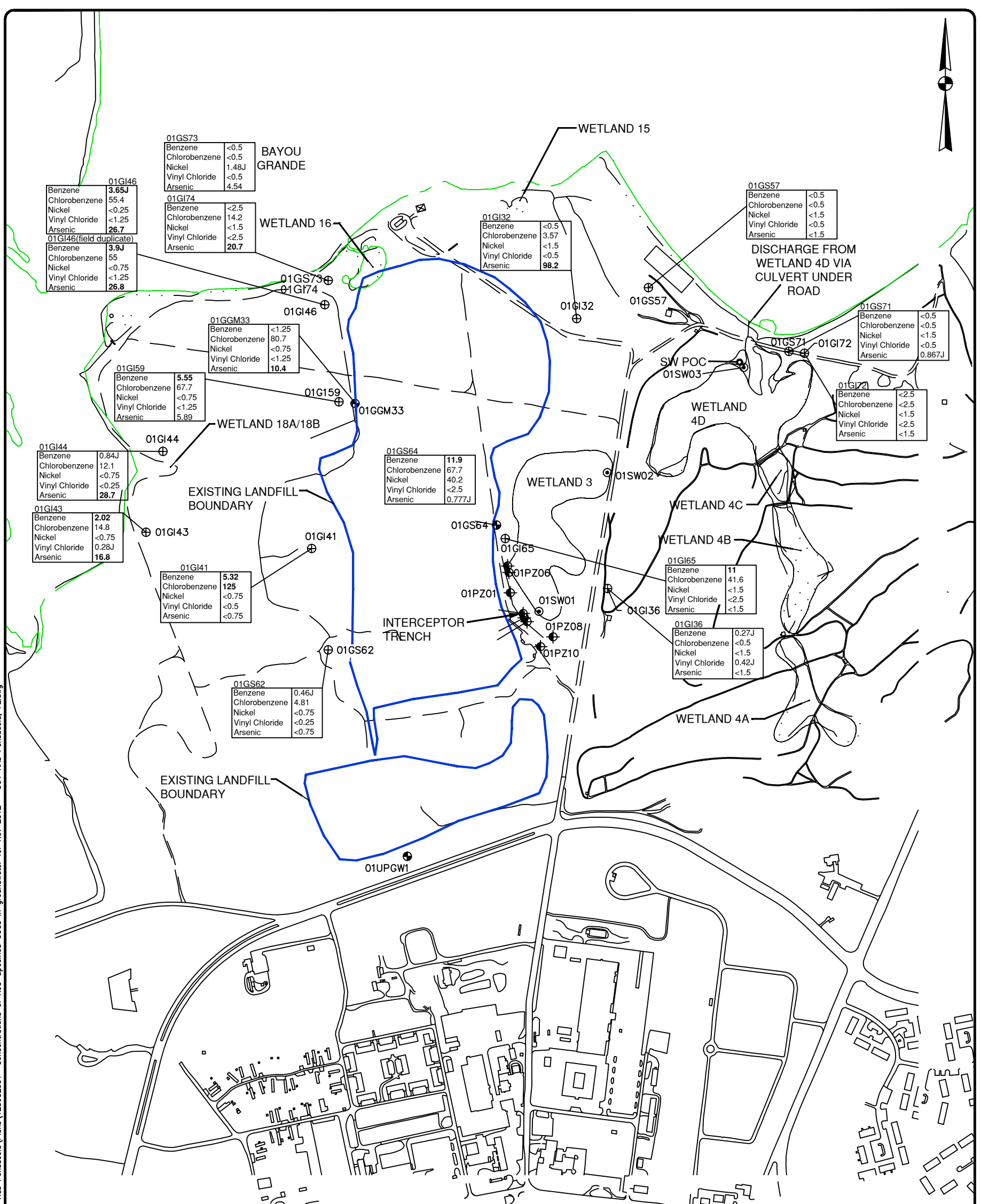
Iron in Piezometers Data Assessment

Groundwater samples were collected from eight piezometers (some of which were previously designated treatment system monitoring locations — 01PZ06, 01PZ07, 01PZ08, and 01PZ10) during the November 2012 sampling events. During the November 2012 sampling event, a groundwater sample was not collected from upgradient well (01UPGW1) because it was dry. Because the upgradient well has been dry since May 2010, a new upgradient well will be installed as part of the activities under the SAP. The analytical results for total iron of the samples collected in May and November 2012 at the sampling piezometers are included in Table 10-2. Previous reports (Tetra Tech 2008) included a comparison between the piezometers located upgradient and downgradient of the treatment system to monitor the effects. However, the treatment system was decommissioned in May 2010. During these sampling events only one piezometer pair representing upgradient/downgradient (PZ10/PZ08) of the treatment system were sampled. The November 2012 analytical result for iron at the sampling location (01PZ10) upgradient of the interceptor trench was 426 µg/L, below the surface water quality criteria (1,000 µg/L) and the freshwater background value (2,360 µg/L). The November 2012 analytical results for iron at the sampling locations 01PZ06, 01PZ07, and 01PZ08 downgradient of the treatment system was 34,700, 24,600 and 1,160 µg/L, respectively.

The November 2012 iron concentrations for downgradient sample location 01PZ06 and 01PZ07 were above the surface water quality criteria and the freshwater background value, The November 2012 iron concentration for downgradient sample location 01PZ08 was above the surface water quality criteria but below the freshwater background value in the November 2012 samples.



\\Esdanet\cad\CAD PROJECTS\12959 Nas Pensacola\Plans\12959E001 Concentrations of Rod-Specified COCs in groundwater for Nov 2012 - 001 NAS Pensacola, FL.dwg



SOURCE: CONCEPTUAL REMEDIATION DESIGN AT OPERABLE UNIT 1
NAVAL AIR STATION PENSACOLA REVISION 1, SEPTEMBER 1, 1998

ROD-Specified Groudwnwater COCs and Corresponding Performance Criteria	
Contaminant	Performance Criteria (ug/L)
Benzene	1
Chlorobenzene	100
Nickel	100
Vinyl Chloride	1
Arsenic	10

- LEGEND
- EXISTING SHALLOW MONITORING WELL LOCATION AND DESIGNATION
01GS42
 - ⊕ EXISTING INTERMEDIATE MONITORING WELL LOCATION AND DESIGNATION
01GS65
 - COASTLINE/SURFACE WATER FEATURE
 - UNPAVED ROAD
 - LANDFILL BOUNDARY
 - ⊕ PIEZOMETER LOCATION
 - ⊙ SURFACE WATER LOCATION
 - BOLD** EXCEEDS BOTH MCL CRITERIA AND THE REFERENCE CONCENTRATION
 - ug/L MICROGRAMS PER LITER
 - SW POC SURFACE WATER POINT OF COMPLIANCE
 - < UNDETECTED AT THE SHOWN VALUE
 - J ESTIMATED VALUE



FIGURE 10-3
COCs IN GROUNDWATER - NOVEMBER 2012
OPERABLE UNIT 1 UFP-SAP
NAVAL AIR STATION PENSACOLA
PENSACOLA, FLORIDA

REQUESTED BY: D.MYERS	DATE: 10/29/13
DRAWN BY: E.ROGERS	DWG NAME: 12959E001



Table 10-2
Detected Concentrations of Iron in Piezometers
Operable Unit 1
Naval Air Station Pensacola, Pensacola, Florida

Piezometer ID	Screened Interval	Upgradient/ Downgradient of Former Treatment System	Nov. 2001	May 2002	Oct. 2002	June 2003	Nov. 2003	June 2005	Dec. 2005	May 2010	Nov. 2010	Aug. 2011	Jan. 2012	Nov. 2012
01PZ01	not known	Downgradient	NI	NI	NI	NI	NI	NI	NI	46,300	35,500	24,500	21,400	15,800
01PZ06	7-12	Downgradient	14,600	18,300	5,680	19,200	36,200	35,000	16,800	28,600	19,600	7,530	16,000	34,700
01PZ06A	35-40	Downgradient	NI	NI	NI	NI	NI	NI	952	820	1,350	1,000	901	1,010
01PZ07	21-26	Downgradient	1690	8,120	4,360	17,500	5,200	45,600	46,800	11,200	9,830	27,600	14,600	24,600
01PZ07A	9-14	Downgradient	NI	NI	NI	NI	NI	NI	78,100	17,900	16,800	25,400	12,800	20,200
01PZ07B	35-40	Downgradient	NI	NI	NI	NI	NI	NI	NI	1,090	1,070	1,040	896	1,080
01PZ08	21-26	Downgradient	NI	NI	2,560	2,620	4,410	3,460	1870	2,940	1,170	1,560	846	1,160
01PZ10	21-26	Upgradient	5,630	1,800	3,260	1,900	2,810	1,920	2,670	1,740	945	671	846	426

Notes:

All concentrations are in micrograms per liter (µg/L) or parts per billion (ppb)

Class III Freshwater Surface Water Criteria from Chapter 62-302.520, Florida Administrative Code (FAC) is 1,000 µg/L.

The applicable reference concentration for iron in freshwater surface water is 2,360 µg/L.

Results in bold are above both the surface water criteria and the reference concentration.

NI = not installed



Table 10-3 Iron Detected in Surface Water Operable Unit 1 NAS Pensacola, Pensacola, Florida															
Seep (01SW001)															
01W0101	01W0102	01W00103	01W00104	01W00105	01W00106	01W00107	OU1-W001-06	OU1-O1W01-12	OU1-01W01-07	OU1-01W01-08	01SW01-0510	01SW01-1110	01SW01-0811	01SW01-0112	01SW01-1112
20-Mar-00	18-Aug-00	23-May-01	1-Nov-01	1-May-02	8-Oct-02	9-Jun-03	17-Nov-03	1-Jun-05	17-Dec-05	8-Feb-06	13-May-10	6-Nov-10	31-Aug-11	17-Jan-12	5-Nov-12
28,000	700,000	9,190	6,710	7,930	7,310	42,400	39,800	5,470	8,900	19,300	1,690/4,840 (DUP)	5,220	NS	NS	8,030
Stream (01SW002)															
01W0201	01W00202	01W00203	01W00204	01W02	01W00206	01W00207	OU1-W002-06	OU1-O1W02-12	OU1-01W02-07	OU1-01W02-08	01SW02-0510	01SW02-1110	01SW02-0811	01SW02-0112	01SW02-1112
20-Mar-00	18-Aug-00	23-May-01	1-Nov-01	30-Apr-02	8-Oct-02	9-Jun-03	17-Nov-03	1-Jun-05	17-Dec-05	8-Feb-06	13-May-10	6-Nov-10	31-Aug-11	17-Jan-12	5-Nov-12
19,500	1,800,000	57,300	36,800	816,000	19,700	1,570,000	15,600	5,620	4,350	11,000	6,890	7,050	12,900	6,500/5,840 (DUP)	5,850
Point of Compliance (01SW03)															
01SW03-0510	01SW03-1110	01SW03-0811	01SW03-0112	01SW03-1112											
13-May-10	6-Nov-10	31-Aug-11	17-Jan-12	5-Nov-12											
345	427	265/291 (DUP)	347	580											

Notes:
All concentrations are in micrograms per liter (µg/L) or parts per billion (ppb).
Class III Freshwater Surface Water Criteria from Chapter 62-302.520, Florida Administrative Code (FAC) is 1,000 µg/L.
The applicable reference concentration for iron in freshwater surface water is 2,360 µg/L.
Results in bold are above both the surface water criteria and the reference concentration.
NS = Not sampled



Table 10-4 Detected Concentrations of COCs in Groundwater Operable Unit 1 Naval Air Station Pensacola, Pensacola, Florida						
Well ID	Date	Benzene	Chlorobenzene	Vinyl chloride	Arsenic	Nickel
Criteria		1	100	1	10	100
01GS57	7/93	ND	ND	ND	ND	158 J
	7/94	NS	NS	NS	NS	NS
	3/00	ND	ND	ND	NA	ND
	8/00	ND	ND	ND	NA	ND
	5/01	ND	ND	ND	NA	ND
	11/01	ND	ND	ND	NA	ND
	5/02	ND	ND	ND	NA	ND
	10/02	ND	ND	ND	NA	ND
	6/03	ND	ND	ND	NA	ND
	11/03	ND	ND	ND	NA	ND
	5/10	ND	ND	ND	NA	1.13 J
	11/10	ND	ND	ND	NA	ND
	8/11	ND	ND	ND	NA	ND
	1/12	ND	ND	ND	NA	ND
	11/12	ND	ND	ND	ND	ND
01GS62	7/93	12	6 J	ND	ND	43.2
	7/94	12	7 J	ND	ND	ND
	3/00	20	15	1.3	NA	ND
	8/00	19	15	1.1	NA	ND
	5/01	21	18.8	ND	NA	1.6 J
	11/01	19.2	23	0.94 J	NA	1.2 J
	5/02	18	20	ND	NA	ND
	10/02	17	25	ND	NA	ND
	6/03	14	28.6	ND	NA	ND
	11/03	16	40	ND	NA	ND
	5/10	ND	0.38 J	ND	NA	0.849 J
	11/10	ND	ND	ND	NA	ND
	8/11	ND	2.43	ND	NA	ND
	1/12	ND	4.62	ND	NA	ND
	11/12	0.46	4.81	ND	ND	ND
01GS64	7/93	13	120	ND	ND	63.4
	7/94	3 J	31	ND	ND	ND
	3/00	NS	NS	NS	NA	NS
	8/00	NS	NS	NS	NA	NS
	5/01	1.9	10	1.4	NA	13.8 J
	11/01	2.4	8.5	1.9	NA	5.6 J
	5/02	1.8 J	8.3	ND	NA	ND
	10/02	5.1	22	ND	NA	13.0
	6/03	7.4	89	ND	NA	24.5
	11/03	6.0	31	ND	NA	22.3
	5/10	1.45	16.5	1 J	NA	45.1
	11/10	ND	13.30	ND	NA	58.5
	8/11	6.65	68	ND	NA	56.3
	1/12	4.94	54.4	ND	NA	50.2
	11/12	11.9	67.7	ND	0.777 J	40.2



Table 10-4 Detected Concentrations of COCs in Groundwater Operable Unit 1 Naval Air Station Pensacola, Pensacola, Florida						
Well ID	Date	Benzene	Chlorobenzene	Vinyl chloride	Arsenic	Nickel
Criteria		1	100	1	10	100
01GS71	7/93	NS	NS	NS	NS	NS
	7/94	ND	ND	ND	ND	ND
	3/00	ND	ND	ND	NA	ND
	8/00	ND	ND	ND	NA	ND
	5/01	ND	ND	ND	NA	ND
	11/01	ND	ND	ND	NA	ND
	5/02	ND	ND	ND	NA	ND
	10/02	ND	ND	ND	NA	ND
	6/03	ND	ND	ND	NA	ND
	11/03	ND	ND	ND	NA	ND
	5/10	ND	ND	ND	NA	2.02 J
	11/10	ND	ND	ND	NA	0.88
	8/11	ND	ND	ND	NA	ND
	1/12	ND	ND	ND	NA	0.897 J
	11/12	ND	ND	ND	0.867 J	ND
01GGM33	7/93	3 J	35	ND	41.7 J	ND
	7/94	2 J	31	ND	16.4	ND
	3/00	ND	3.8	ND	NA	ND
	8/00	ND	4.7	ND	NA	ND
	5/01	0.75 J	22.6	ND	NA	ND
	11/01	ND	7.7	ND	NA	ND
	5/02	ND	19	ND	NA	ND
	10/02	5	52	ND	NA	ND
	6/03	1.3	70.1	ND	NA	ND
	11/03	0.8 J	66	ND	NA	ND
	5/10	2.62	76.4	0.701 J	NA	ND
	11/10	3.23	109	0.56	NA	ND
	8/11	1.82	1.79	ND	NA	ND
	1/12	0.991 J	81.1	0.595 J	NA	ND
	11/12	ND	80.7	ND	10.4	ND
01UPGW1	7/93	NS	NS	NS	NS	NS
	7/94	NS	NS	NS	NS	NS
	3/00	NS	NS	NS	NS	NS
	8/00	NS	NS	NS	NS	NS
	5/01	NA	NA	NA	NA	NA
	11/01	ND	ND	ND	NA	ND
	5/02	ND	ND	ND	NA	1.6
	10/02	ND	ND	ND	NA	ND
	6/03	ND	ND	ND	NA	ND
	11/03	NS	NS	NS	ND	NS
	5/10	ND	ND	1	NA	9.83
	11/10	NS	NS	NS	NS	NS
	8/11	NS	NS	NS	NS	NS
	1/12	NS	NS	NS	NS	NS
	11/12	NS	NS	NS	NS	NS



Table 10-4 Detected Concentrations of COCs in Groundwater Operable Unit 1 Naval Air Station Pensacola, Pensacola, Florida						
Well ID	Date	Benzene	Chlorobenzene	Vinyl chloride	Arsenic	Nickel
Criteria		1	100	1	10	100
01GI32	7/93	2 J	4 J	ND	55.2 J	32
	7/94	NS	NS	NS	NS	NS
	3/00	1.6	6.3	3	NA	ND
	8/00	0.82 J	3.5	1.5	NA	ND
	5/01	0.86 J	4.3	1.9	NA	ND
	11/01	0.67 J	ND	1.2	NA	ND
	5/02	ND	5J	ND	NA	ND
	10/02	0.88 J	2.5	0.7 J	NA	ND
	6/03	0.78 J	4.8	ND	NA	ND
	11/03	1	7	1	NA	ND
	5/10	ND	10.7	2.06 J	NA	ND
	11/10	ND	9.83	0.71	NA	ND
	8/11	ND	5.66	0.526 J	NA	ND
	1/12	ND	6.04	0.689 J	NA	ND
	11/12	ND	3.57	ND	98.2	ND
01GI36	7/93	2 J	ND	ND	11.7	219 J
	7/94	ND	ND	ND	ND	NA
	3/00	0.6	ND	3.7	NA	ND
	8/00	0.85 J	ND	3.9	NA	ND
	5/01	0.79 J	ND	3.6	NA	ND
	11/01	ND	ND	3.0	NA	ND
	5/02	ND	ND	ND	NA	ND
	10/02	0.59 J	ND	2.2 J	NA	ND
	6/03	ND	ND	ND	NA	ND
	11/03	0.3 J	ND	1 J	NA	ND
	5/10	ND	ND	1 J	NA	ND
	11/10	ND	ND	0.37	NA	ND
	8/11	ND	ND	ND	NA	ND
	1/12	ND	ND	ND	NA	ND
	11/12	0.27 J	ND	0.42 J	ND	ND
01GI41	7/93	74	120	ND	26	24.1
	7/94	80	110	3	ND	NA
	3/00	69	130	6.4	NA	ND
	8/00	57	95	3.3	NA	ND
	5/01	33.1	49.6	4.2	NA	ND
	11/01	32.7	47.4	6.8	NA	ND
	5/02	18	68	2.2 J	NA	ND
	10/02	22	48	1.4	NA	ND
	6/03	27.7	86.6	0.66 J	NA	ND
	11/03	31	100	ND	NA	ND
	5/10	10.9	173	0.3 J	NA	ND
	11/10	13.4	124	ND	NA	ND
	8/11	16.2	148	0.403 J	NA	ND
	1/12	0.298 J	49.9	ND	NA	ND
	11/12	5.32	125	ND	ND	ND



Table 10-4 Detected Concentrations of COCs in Groundwater Operable Unit 1 Naval Air Station Pensacola, Pensacola, Florida						
Well ID	Date	Benzene	Chlorobenzene	Vinyl chloride	Arsenic	Nickel
Criteria		1	100	1	10	100
01G143	7/93	3 J	2 J	ND	36.4 J	ND
	7/94	NS	NS	NS	NS	NS
	3/00	NS	NS	NS	NS	NS
	8/00	6.6	10	2.3	NA	ND
	5/01	7.7	13.3	1.7	NA	ND
	11/01	5.6	11.5	2	NA	ND
	5/02	5.2	12	1.9 J	NA	ND
	10/02	7.2	14	3.4	NA	ND
	6/03	5	12.8	1.7	NA	ND
	11/03	6	15	ND	NA	ND
	5/10	4.87	27.3	1.28 J	NA	ND
	11/10	4.08	24.10	0.68	NA	ND
	8/11	2.75	20.4	0.579 J	NA	ND
	1/12	2.36	15.7	0.485 J	NA	ND
	11/12	2.02	14.8	0.28 J	16.8	ND
01G144	7/93	3 J	32	ND	25.5	59.2
	7/94	NS	NS	NS	NS	NS
	3/00	5.3	58	2.6	NA	ND
	8/00	4.1 J	77	4.4 J	NA	ND
	5/01	0.81 J	36.7	ND	NA	ND
	11/01	1.2	36.4	0.96 J	NA	ND
	5/02	ND	27	ND	NA	ND
	10/02	ND	2.2	ND	NA	ND
	6/03	6.3	41.2	0.63 J	NA	ND
	11/03	3	58	1 J	NA	ND
	5/10	6.58	48.5	1.21 J	NA	ND
	11/10	NS	NS	NS	NA	NS
	8/11	NS	NS	NS	NA	NS
	1/12	ND	6.01	ND	NA	ND
	11/12	0.84 J	12.1	ND	28.7	ND
01G146	7/93	3 J	18	19	27	ND
	7/94	3 J	18	12	24	NA
	3/00	2	17	17	NA	ND
	8/00	ND	18	19	NA	ND
	5/01	3.9	33.6	23.2	NA	ND
	11/01	4.2	35.6	25.6	NA	ND
	5/02	3J	29	13	NA	ND
	10/02	5.1	35	42	NA	ND
	6/03	3.3	30.9	27.1	NA	ND
	11/03	4	36	34	NA	ND
	5/10	3.36	48.7	11.3 J	NA	ND
	11/10	1.36	18.50	3.44	NA	ND
	8/11	2.78	40.2	3.16	NA	ND
	1/12	1.69	24.1	0.711 J	NA	ND
	11/12	3.65 J	55.4	ND	26.7	ND



Table 10-4 Detected Concentrations of COCs in Groundwater Operable Unit 1 Naval Air Station Pensacola, Pensacola, Florida						
Well ID	Date	Benzene	Chlorobenzene	Vinyl chloride	Arsenic	Nickel
Criteria		1	100	1	10	100
01GI59	7/93	8	45	ND	33.2 J	ND
	7/94	ND	19	ND	7.5	NA
	3/00	11	55	11	NA	ND
	8/00	9.7	51	7.7	NA	ND
	5/01	10.7	72.4	6.8	NA	ND
	11/01	8.9	72.2	3.6	NA	ND
	5/02	9	66	3.5 J	NA	ND
	10/02	15 J	84 J	1.6 J	NA	ND
	6/03	6.7	87.5	0.59 J	NA	ND
	11/03	5	98	0.3 J	NA	ND
	5/10	3.08	91.10	1	NA	ND
	11/10	3.62	109	0.66	NA	ND
	8/11	5.21	157	ND	NA	ND
	1/12	4.3	104	0.331 J	NA	ND
	11/12	5.55	67.7	ND	5.89	ND
01GI65	7/93	11	7	23	68.8	49.5
	7/94	10	23	7 J	ND	NA
	3/00	15	49	4.9	NA	ND
	8/00	13	39	4.1	NA	ND
	5/01	11.7	40.6	4.4	NA	ND
	11/01	10	37.1	3.4	NA	ND
	5/02	8.3	26	3.5 J	NA	ND
	10/02	11	27	5.5	NA	ND
	6/03	14.4	43.6	2	NA	ND
	11/03	14	38	4	NA	ND
	5/10	11.70	37	4.85	NA	0.844 J
	11/10	12.60	37.40	ND	NA	ND
	8/11	12.1	50.5	2.44	NA	ND
	1/12	13.7	50.2	1.65	NA	ND
	11/12	11	41.6	ND	ND	ND
01GI72	7/93	NS	NS	NS	NS	NS
	7/94	ND	ND	ND	ND	NA
	3/00	ND	ND	ND	NA	ND
	8/00	ND	ND	ND	NA	ND
	5/01	ND	ND	ND	NA	ND
	11/01	ND	ND	ND	NA	ND
	5/02	ND	ND	ND	NA	ND
	10/02	ND	ND	ND	NA	ND
	6/03	ND	ND	ND	NA	ND
	11/03	ND	ND	ND	NA	ND
	5/10	ND	ND	ND	NA	1.23 J
	11/10	ND	ND	ND	NA	ND
	8/11	ND	ND	ND	NA	ND
	1/12	ND	ND	ND	NA	ND
	11/12	ND	ND	ND	ND	ND



Table 10-4 Detected Concentrations of COCs in Groundwater Operable Unit 1 Naval Air Station Pensacola, Pensacola, Florida						
Well ID	Date	Benzene	Chlorobenzene	Vinyl chloride	Arsenic	Nickel
Criteria		1	100	1	10	100
01GS73	5/10	ND	ND	ND	NA	3.16
	11/10	ND	ND	ND	NA	ND
	8/11	ND	ND	ND	NA	5.04
	1/12	ND	ND	ND	NA	1.02 J
	11/12	ND	ND	ND	4.54	1.48 J
1GI74	5/10	1.22	15.10	9.15	NA	1.74
	11/10	0.9	20.50	3.77	NA	ND
	8/11	0.748 J	12.7	5.5	NA	ND
	1/12	0.863 J	16.7	3.76	NA	ND
	11/12	ND	14.2	ND	20.7	ND

Notes:

Concentrations are in micrograms per liter.

COCs = Contaminant of concern

ND = Not detected

NS = Not sampled

NA = Not analyzed for parameter

J = estimated concentration

Bold results are above the GCTL from Chapter 62-777, F.A.C.

Surface Water Data Assessment

Based on the LTM results presented in Table 10-3 since 2000 the concentration of iron detected in surface water samples (seep and stream samples) appear to be highly variable, sometimes changing in concentration by a factor of 100. The data demonstrate that iron continues to be detected in the surface water samples collected from Wetland 3 at concentrations that exceed the Florida Surface Water Quality Criteria of 1,000 µg/L per Chapter 62-302, F.A.C. and the freshwater background concentration of 2,360 µg/L. However, the concentration (580 µg/L) of iron at the surface water point of compliance (SW POC) location (01SW03) is less than the freshwater background concentration of 2,360 µg/L.

The levels of iron associated with Wetland 3 are consistently higher than samples from other Installation Restoration Program (IRP) sites. There is anecdotal evidence of disposal of significant waste that would be iron-rich (for example, engines). In addition, native iron and waste-derived iron are likely being mobilized via reducing conditions within the landfill footprint, and are being used by iron bacteria at discharge points where conditions are aerobic. The weight of evidence in the Wetland 3 area (ORP readings range from -2.4 mV at PZ08 to -125.1 mV at PZ07B, DO readings ranged from 0.15 mg/L at PZ07B to 0.87 mg/L at GI44 in January 2012) indicates that the high iron may be a result of mobilization of leachate from the landfill waste.



Groundwater Data Assessment

Groundwater samples collected from all (15 or 16 depending on the event) monitoring wells at OU 1 were analyzed for three VOCs (benzene, chlorobenzene, and vinyl chloride) and one inorganic (nickel) that were specified in the ROD. The groundwater COC criteria are the Florida GCTLs/EPA MCLs for each parameter. Table 10-4 summarizes the COC detections OU 1 and the November 2012 detections are shown on Figure 10-3.

Benzene was detected in the groundwater samples collected for the November 2012 sampling event at concentrations exceeding its ROD specified criteria in one shallow aquifer zone monitoring well and five intermediate aquifer zone monitoring wells. Benzene concentrations in the groundwater sample collected from the shallow aquifer zone monitoring wells ranged from 0.46 to 11.9 µg/L. Benzene concentrations in the groundwater sample collected from the intermediate aquifer zone monitoring wells ranged from 0.27 to 11 µg/L.

Chlorobenzene was not detected in the November 2012 sampling event at concentrations above its ROD specified criteria in the shallow aquifer zone; however, it was detected in one intermediate aquifer zone monitoring well (01GI41 at 125 µg/L.). Chlorobenzene concentrations in groundwater samples collected from the shallow aquifer zone monitoring wells ranged from concentrations of 4.81 µg/L to 80.7 µg/L. Chlorobenzene concentrations in groundwater samples collected from the intermediate aquifer zone monitoring wells ranged from 3.57 µg/L to 125 µg/L.

Vinyl chloride was detected in two intermediate zone monitoring wells (01GI36 at 0.42 µg/L and 01GI43 at 0.28 µg/L) below its ROD specified criteria of 1 µg/L.

Arsenic was detected in four shallow monitoring wells ranging in concentrations from 0.777 µg/L at 01S64 to 10.4 µg/L at 01GM33. One of the detections was above the current MCL of 10 µg/L. Arsenic was detected in six intermediate zone monitoring wells ranging from 5.89 µg/L at 01GI59 to 98.2 µg/L at 01GI32. Five of the six detections exceed the current MCL.

Nickel was detected in two shallow monitoring wells, 01GS64 at 40.2 µg/L and 0101GS73 at 1.48 µg/L. Both detections were below its ROD specified criteria of 100 µg/L.



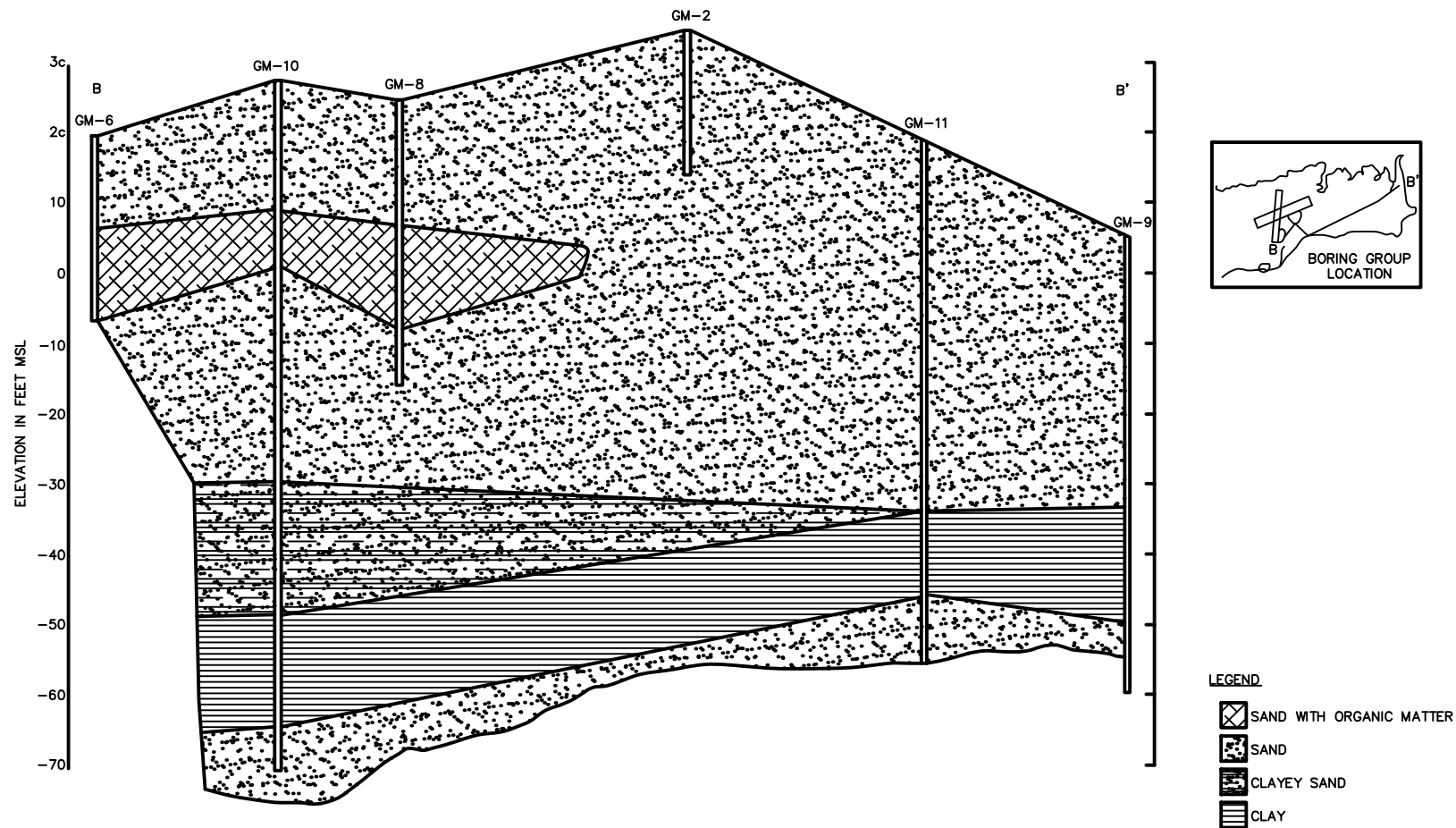
Benzene, chlorobenzene, vinyl chloride, arsenic, and nickel were not detected at concentrations that exceed their Florida Natural Attenuation Default Concentrations (NADCs) in the groundwater samples collected for the November 2012 sampling event.

The data were evaluated to the DQOs established in the previous UFP SAP (Tetra Tech 2010). However, in correspondence dated October 18, 2012, U.S. EPA noted that the three decision rules were a combination of decision rules in the UFP SAP and groundwater monitoring decision rules that were in the Five-Year Review (Tetra Tech 2008). U.S. EPA also noted the benzene, chlorobenzene, and vinyl chloride were above their ROD performance criteria and their GCTLs and that monitoring should continue for benzene, chlorobenzene, and vinyl chloride. Comments from FDEP on the report and on the recommendations have not been received to date.

10.2 Geology and Hydrogeology

The NAS Pensacola facility is surrounded to the south by Pensacola Bay and north by Bayou Grande. The terrain is generally flat with rolling undulations. Undeveloped areas, particular on the west side of the facility, are mostly wooded with pines and hardwoods, with intertidal marshes and salt-tolerant vegetation near the shores; the more developed south-central and eastern portions of the facility contain less native vegetation, especially in training and industrial complexes and on the sprawling A.C. Read Golf Course. The subtropical climate averages 60 inches of rainfall per year, some coming in heavy downpours and occasional hurricanes. The shallow geology consists of sand and silty sand, supporting a shallow, unconfined, highly transmissive surficial aquifer, in which shallow groundwater flow mimics topography.

Stratigraphy in this region consists of Quaternary terrace marine and fluvial deposits underlain by a thick sequence of interlayered fine-grained clastic deposits and carbonate strata of the Tertiary age (Southeastern Geological Society [SEGS 1986]). Three main regional hydrologic units have been defined within this stratigraphic column. In order of increasing depth, these units are: the surficial sand-and-gravel aquifer, the Intermediate System, and the Floridian Aquifer system. Figure 10-4, Geological Cross-Section, presents a generalized cross-section of these units. Regionally, the surficial zone is approximately 30 to 60 feet thick and generally is composed of poorly graded quartz sand. The Intermediate System is composed of gray to blue, sandy and silty marine clay with some shell fragments, and clayey sands with total thickness ranging from 8 to 40 feet.



SOURCE: GERAGHTY & MILLER, 1986

DRAWN BY	DATE
CK	10/06/09
CHECKED BY	DATE
J.D. SPALDING	10/06/09
REVISED BY	DATE
CK	10/06/09
SCALE	
NOT TO SCALE	



FIGURE 10-4
GEOLOGICAL CROSS SECTION OF THE
SURFICIAL AQUIFER
OPERABLE UNIT 1 UFP-SAP
NAVAL AIR STATION PENSACOLA
PENSACOLA, FLORIDA

CONTRACT NO.
JM40

OWNER NO.

APPROVED BY DATE
A.HARRIS 10/16/13

DRAWING NO. REV.
FIGURE 10-4 0



At OU 1, the surficial aquifer is subdivided into the surficial zone (approximately 30 to 60 feet thick, which is underlain by the lower permeability zone (ranging in thickness from 8 to 40 feet), and the main producing zone (estimated to be about 300 feet thick). The surficial aquifer is composed of unconsolidated clastic deposits (Wilkins et al. 1985). The stratigraphic relationship of these zones within the surficial aquifer is illustrated on Figure 10-4.

Groundwater of the surficial aquifer is classified by U.S. EPA as IIA and by FDEP as G-2, which designates the groundwater as a potential source of drinking water. The main-producing zone is generally the most permeable and is the principal water supply source for the Pensacola area.

Groundwater in this zone generally occurs under confined conditions, and recharge to the zone occurs primarily by leakage through the low permeability zone. NAS Pensacola derives potable water from main-producing zone wells at Corry Field, located approximately three miles north of OU 1.

At NAS Pensacola, assessment wells have been completed in the surficial zone, the low permeability zone, and the upper portion of the main producing zone. Although previous studies have defined shallow, intermediate, and deep groundwater "zones", these refer to three general depths at which monitoring wells have been completed (shallow = approximately 25 ft deep [straddling the water table], intermediate = 45 ft deep [screened immediately above a marine clay marking the top of the low permeability zone], and deep = 70 ft [immediately below the base of the marine clay, marking the top of the main producing zone]).

10.3 Conceptual Site Model (CSM)

The OU 1 landfill material is a potential contaminant source for landfill soils, for groundwater beneath and downgradient of the landfill, and for surface water and sediments downgradient of the landfill. Chemical contamination has not been detected in soil outside the footprint of the landfill, but groundwater contamination has been detected beyond the footprint of the landfill.

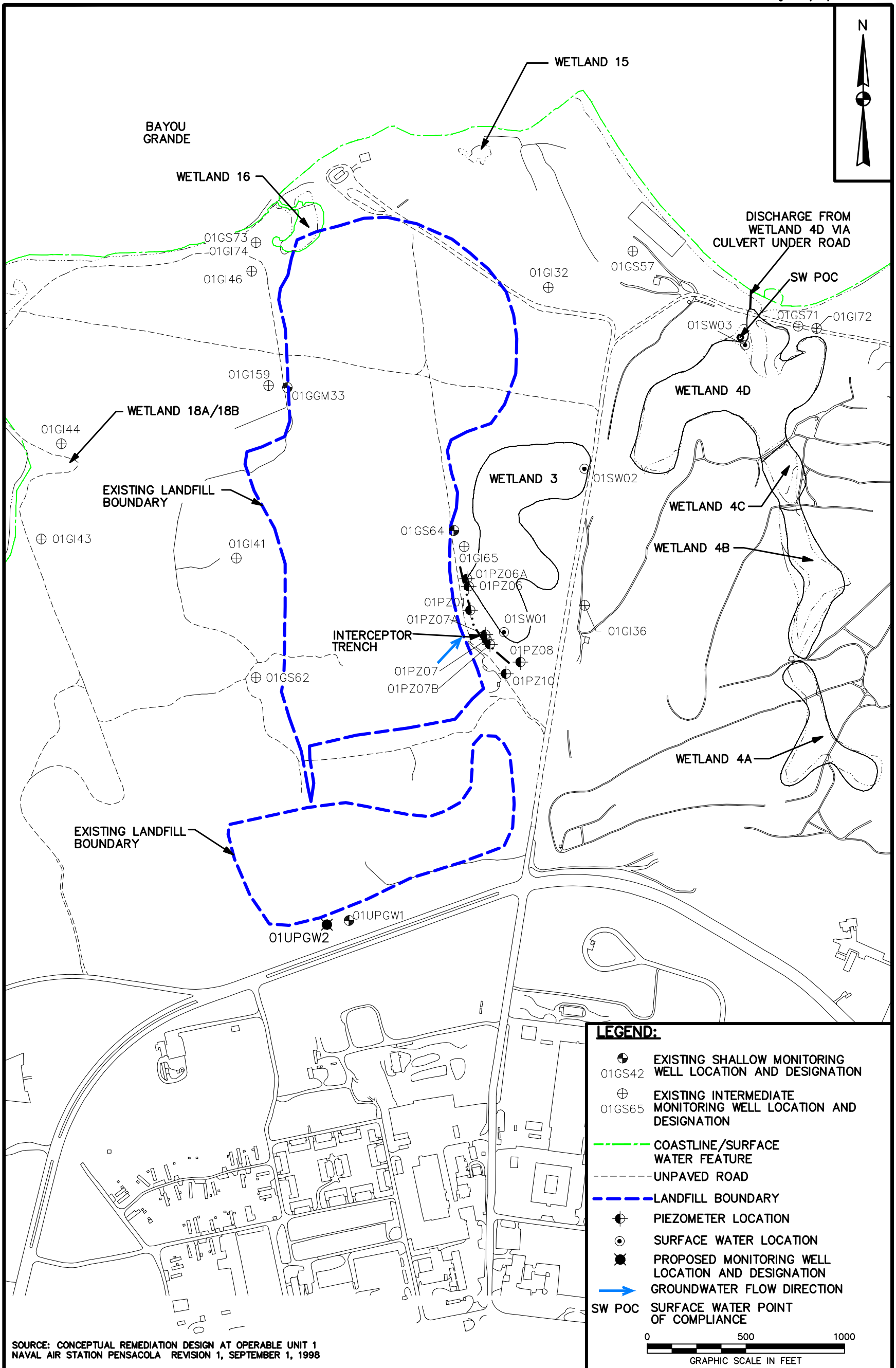
The fill materials vary considerably in chemical composition and are heterogeneously distributed; therefore, the U.S. EPA presumptive remedy (e.g., capping with monitoring) has been applied to this site (U.S. EPA Presumptive Remedies for Municipal Landfill Sites April 1992). The cap is a simple, vegetated soil cover. Land use controls (LUCs) are used to prevent unauthorized access to OU 1.



Elevated concentrations of select VOCs and metals have been detected in groundwater under and around the landfill. Aerobic oxidation of the organic volatiles (vinyl chloride and benzene) cannot be definitively concluded as occurring at OU 1 at this time, but evidence for this process does exist. Contaminants persist in the wells located at intermediate depths within the surficial aquifer. However, where oxidizing conditions are present in either the shallow or intermediate intervals of the surficial aquifer, there is little evidence of contamination. For example, vinyl chloride and benzene concentrations are persistent in well 01GI41 that is under reducing conditions, but the shallow well in the same cluster (01GS42) has oxidizing conditions and these contaminants are not detectable in that well. The ROD documents that no deep (main-producing zone) contamination has been detected.

Groundwater flow from the landfill is radial to the west, north, and east with predominant flow to the north and northeast toward Bayou Grande. The existing shallow and intermediate wells, as shown on Figure 10-5, were chosen for sampling based on the local hydrogeology as described in Section 10.2. Specifically, existing shallow and intermediate wells were chosen in the path of likely groundwater contamination. Further details regarding existing wells including well screen depth and depth to groundwater can be reviewed within the most recent monitoring event, 2011 Annual Groundwater Monitoring Report for Long-Term Monitoring, Operable Unit 1 (Tetra Tech, *in press*). One upgradient groundwater monitoring well, 01UPGW1, is located outside the landfill's radial influence near, but to the south of, the landfill (Figure 10-5), Proposed Groundwater Sampling Location Map); however, groundwater samples have not been collected from this well since May 2010 because the well has been dry. A new upgradient groundwater monitoring well, 01UPGW2, is proposed to monitor background concentrations. Critical Conceptual Site Model (CSM) transport components include: Chlorobenzene and vinyl chloride contamination in groundwater are markers for the maximum extent of ROD COCs.

Anoxic groundwater (showing limited aerobic degradation of chlorobenzene and vinyl chloride) passes through a mixing zone at an aquifer-surface water body interface (for example, Wetland 3), and at this point the geochemistry changes naturally to an oxygen rich environment. The aerobic microbial population present in this zone (including streambed sediments) has been shown to create rapid mineralization of these two compounds, resulting in their degradation to more innocuous compounds. It is believed that this mineralization is the reason chlorobenzene and vinyl chloride has been detected infrequently at OU 1. A slight downward hydraulic gradient, coupled with less oxygenated conditions, could explain the slightly greater contaminant concentrations in the intermediate groundwater zone. Oxidative destruction of VOCs is expected greater in the more oxygenated shallow zone.



SOURCE: CONCEPTUAL REMEDIATION DESIGN AT OPERABLE UNIT 1
NAVAL AIR STATION PENSACOLA REVISION 1, SEPTEMBER 1, 1998

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FIGURE 10-5
PROPOSED SAMPLING
LOCATION MAP
OPERABLE UNIT 1 UFP-SAP
NAVAL AIR STATION PENSACOLA
PENSACOLA, FLORIDA

CONTRACT NO.	JM40
OWNER NO.	
APPROVED BY	DATE
A.HARRIS	03/14/14
DRAWING NO.	REV.
FIGURE 10-5	0



Dilution and volatilization assist in the reduction of contaminants at the groundwater aquifer/surface water interface. Mixing occurs at most interfaces, but is enhanced where the aquifer discharges into a tidally influenced surface water body. Tidal pumping (groundwater table fluctuations) at the aquifer/surface water interface created by the change in tide level introduces a cyclic yet consistent source of oxygen and continuously dilutes the concentrations in this mixing zone. With this source of oxygen, the VOCs (e.g., vinyl chloride and benzene) will volatilize and will therefore be reduced in concentration.

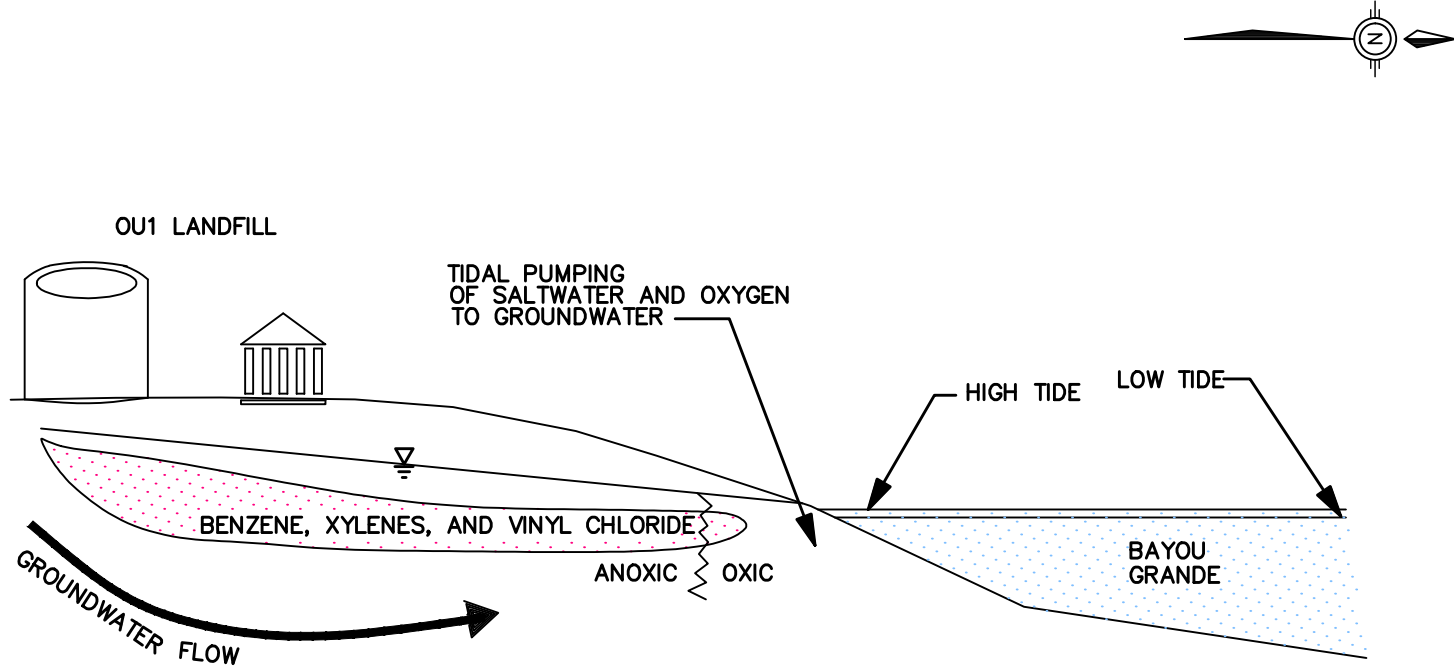
No elevated VOC concentrations have been detected in surface water or sediment samples collected around the landfill. Presumably, if any VOCs have migrated into surface water they have volatilized or mineralized. Other organic compounds and metals were detected most frequently and at highest concentrations in wetland sediments and in Bayou Grande sediments, indicating that the

wetland sediments are effectively binding these materials. Iron contamination has been detected in surface water, in seeps emanating from the east side of the landfill, and in sediments around the landfill. Wetland 3 exhibits elevated iron concentrations leached from the landfill, but the ITS installed between the landfill and Wetland 3 to prevent iron migration was ineffective at reducing the iron concentrations to less than the applicable performance criteria. Wetland 3, by its proximity to the landfill, is considered to be an extension of the landfill. Wetland 4D, located east and downgradient of Wetland 3, has not exhibited any impacts from landfill contamination. Wetland 4D discharges to Bayou Grande, which has been classified by FDEP as a Class III water body, indicating its use for recreation and maintaining a well-balanced fish and wildlife population.

The CSM for OU 1 is presented on Figure 10-6.

10.4 Exposure Pathways and Potential Receptors

Current LUCs prevent construction on the landfill and the current cap prevents exposure of occupational workers, visitors, maintenance workers, and trespassers to contaminated soil. Furthermore, groundwater is not currently used for drinking water so no current human exposure to groundwater is anticipated. Hypothetical future human receptors could include a future resident who might drink groundwater, swim in Bayou Grande, or recreate in one of the downgradient wetlands. Pathways of exposure in these cases would include incidental ingestion, dermal contact with contaminated soil, groundwater, and surface water, and ingestion of groundwater water (as drinking water). Other receptors are also possible, but the resident is considered to represent the greatest potential exposure and hence is the only future risk scenario to be considered. The decision criteria for this project are designed to be protective of all potential receptors.



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FIGURE 10-6
NATURAL ATTENUATION CONCEPTUAL
SITE MODEL
OPERABLE UNIT 1 UFP-SAP
NAVAL AIR STATION PENSACOLA
PENSACOLA, FLORIDA

CONTRACT NO.
JM40

OWNER NO.

APPROVED BY
A.HARRIS

DATE
10/16/13

DRAWING NO.
FIGURE 10-6

REV.
0



Ecological receptors at OU 1 include a variety of plants, invertebrates, mammals, and avian species. Ecological receptors, however, are not exposed to groundwater which is the environmental medium of primary concern, except where groundwater seeps into surface water bodies such as Wetland 3. Wetlands associated with OU 1 (Wetlands 4D, 15, and 18) are being assessed in OU 16. Wetland 3 is being assessed for its ability to be used as a treatment system for iron in lieu of the ITS.

There are no known cultural concerns associated with this landfill.



SAP WORKSHEET #11: PROJECT QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS STATEMENTS

(UFP-QAPP Manual Section 2.6.1)

The following text describes the development of the Project Quality Objectives (PQOs) using U.S. EPA's DQO (System Planning) Process.

11.1 Problem Statements

Based on the CSM, the problem definition is two-fold:

- The existing ROD states that Performance Standards [the federal Maximum Contaminant Levels (MCLs), or the Florida Primary Drinking Water Standard, whichever is lower] for the four contaminants including nickel, benzene, chlorobenzene, and vinyl chloride (ROD-specified COCs) listed in the ROD must be achieved for groundwater contaminated by the landfill. Arsenic is being added as a chemical by ROD Amendment because of a reduction in the MCL from 50 µg/L to 10 µg/L. A monitoring plan must be developed to establish whether ROD requirements are being satisfied, or whether any additional actions are needed to limit the impact of the landfill on groundwater.
- Because iron has been detected flowing into Wetland 3 at concentrations greater than the Florida Surface Water Cleanup Target Level (SWCTL) criterion of 1,000 µg/L, there is a concern that the Bayou Grande ecology could be at risk from iron contamination. Therefore, to protect Bayou Grande from this contamination, iron concentrations at the SW POC immediately upgradient (south) of Bayou Grande must be monitored to determine whether iron concentrations exceed the U.S. EPA- and FDEP-approved background threshold. This value was determined in a Technical Memorandum to be twice the mean background concentration at the site, which is 5,862 µg/L (Tetra Tech 2009).

According to the Navy policy for groundwater monitoring optimization, long-term groundwater monitoring may only be reduced when contaminant levels have fallen to concentrations that are less than the regulatory action levels and are no longer a threat to local water supply wells. Optimization of the groundwater monitoring program is expected to occur over time, in accordance with the recently updated Navy guidance document, *Guidance for Planning and Optimizing Monitoring Strategies* (Battelle 2008). Annual data reviews will consider the potential for optimization. This will allow the Navy to limit the chemicals and wells included in the program at the earliest possible time, and to tailor the monitoring frequency to make optimal use of monitoring data. The Navy also expects that LUCs will remain in place for at least as long as the landfill exists.



Impacts to sediment and soil are not a concern for this project, since it has already been demonstrated in the RI Report that sediment is not impacted (EnSafe 1996). Impacts to surface water are limited to iron; therefore, investigation of other contaminants in surface water is not necessary.

11.2 Identify the Goals of the Study

The ultimate goal of this study is to provide information needed to complete the Annual Remedy Effectiveness Report for OU 1 and for the optimization of the monitoring program.

11.3 Inputs to Problem Resolution

To resolve the problem statement above, the following information is needed. The ROD-specified parameters including arsenic will be monitored as long as the four parameter concentrations exceed the ROD-specified criteria. Adherence to the ROD is prescribed by law.

The following physical and chemical data will be collected during this investigation:

Groundwater Monitoring Criteria and Parameters:

- Groundwater concentrations for the ROD parameters including arsenic are listed in Section 10.3. Metals will be analyzed for total only (not for dissolved metals).
- Performance Criteria for ROD parameters including arsenic.
- Site-wide background concentrations for ROD (including arsenic) metals. This information will not be used initially in the monitoring program, but is expected to be incorporated into future decision rules (see Section 11.4) to account for the potential that background/upgradient concentrations are greater than the Performance Criteria.

Surface Water Monitoring Criteria and Parameters:

- Surface water concentrations for total iron.
- Marine surface water background concentration threshold of 5,862 µg/L (as documented in the Technical Memorandum, Tetra Tech, 2009), based on two times the mean background iron concentration obtained in representative reference marine wetland locations on site.



- Fresh surface water background concentration threshold of two times the mean background concentration of 2,360 µg/L, as documented in the OU 16 (Site 41) Final Remedial Investigation Report, Site 41 — Operable Unit 16 — NAS Pensacola Wetlands (EnSafe 2007), obtained in representative reference freshwater wetland locations on site.

Additional Parameters:

- Natural attenuation parameters for surface water and groundwater. As part of the optimization strategy inclusion of natural attenuation parameters to verify or support an evaluation of why the contaminant concentrations are decreasing may be useful and should be considered during the optimization evaluations.
- Groundwater well stabilization parameters: dissolved oxygen (DO), ORP, pH, specific conductance, temperature, and turbidity by field instrument.
- Synoptic groundwater levels. These levels shall be measured to a precision of 0.01 foot within a 24-hour period in all wells being monitored to ensure that the measurements are representative of the same time frame. These measurements shall not be made sooner than 24-hours after a significant precipitation event, as decided by the TOM.
- Surface water levels in Wetlands 3 and 4D. The levels of surface water shall be measured to the nearest 0.01 foot at all existing wetland staff gauges.

To conduct comparisons of site data to the Project Action Levels (PALs) for groundwater and surface water, the selected laboratory should be able to achieve Quantitation Limits (QLs) that are low enough to measure constituent concentrations at less than the PALs. The PALs are identified on Worksheet #15. The Project Team will accept the laboratory analytical results between the detection limit (DL) and the QL if the results are "J" qualified. J-flagged data will be accepted to achieve project goals when the PAL is between the QL and the DL. When the PAL is less than the DL for a particular analyte or analytes, an evaluation of DLs and the impact on data usability will be discussed in the data report. Any limitations on the data will be documented at that time and, if significant data gaps remain, additional data will be collected.

Quality control samples for estimating precision, bias, and contamination potential will be collected at frequencies established in Worksheet #12.



11.4 Define the Study Boundaries

Groundwater of interest is the shallow and intermediate groundwater zone of the surficial aquifer downgradient from the landfill that could be affected by contaminants leaching from the landfill. As a point of reference, the upgradient well is also of interest for metals concentrations. Figure 10-5, Groundwater Sampling Location Map, shows a general area of the primary landfill, monitoring well locations, and the groundwater interceptor trench between the landfill and Wetland 3.

The SW POC is near the northeastern edge of Wetland 4D, immediately before this wetland discharges to Bayou Grande. The basis for the location of this SW POC is two-fold. One is that Wetland 3 is considered to be an extension of the OU 1 landfill, therefore it would not serve as an appropriate SW POC. The other is that the selected SW POC is protective of Bayou Grande, which is a Class III surface water body requiring protection. A particular location for the SW POC was identified in a Technical Memorandum (Tetra Tech 2009).

Pending approval of the modified groundwater monitoring program in the OU 1 2011 Annual Groundwater Monitoring Report (Tetra Tech, *in press*), the groundwater and surface water monitoring frequency will be semi-annual. The potential to lengthen the time between sampling rounds will be considered during annual monitoring program evaluation.

Low tide was considered to yield optimal concentrations for surface water sampling or groundwater sampling near Bayou Grande. Biasing toward low tide is expected to yield the highest contaminant concentrations for the tidal cycle. This will ensure that no contamination is overlooked and that results are not biased low. Further inland, the effects of tidal fluctuations on groundwater levels are minimal. Contaminant concentrations should be measured at all groundwater monitoring wells at approximately the same time to ensure that the same time frame is represented by the samples.

No particular season has been identified as more optimal for sampling.

Surface water sampling locations upgradient (south) of the SW POC will be monitored to ensure that increasing concentrations indicative of a potential increase at the SW POC are detected early enough to prevent an increase at the SW POC. Recommendations for groundwater optimization will be made annually (in annual monitoring reports) based on the latest Navy groundwater monitoring optimization guidance.



11.5 Analytical Approach

As part of the UFP-QAPP (U.S. EPA 2005) process, decision rules were determined and approved by the Project Stakeholders prior to the initiation of the LTM fieldwork. The Decisions Rules include:

Decision Rule #1:

Determine whether any contaminant concentration in groundwater exceeds the Performance Criteria (for the four ROD-specified contaminants) for the two most recent rounds of monitoring. If any ROD-specified contaminant concentration does not exceed the Performance Criteria in both rounds, the planning team will convene to optimize the monitoring strategy (i.e., reduce sampling frequency, reduce number of wells). If all contaminants exceed the Performance Criteria in at least one of the two rounds, no change will be made to the monitoring program and LUCs.

Decision Rule #2:

Determine whether iron concentrations in surface water exceed the background threshold value of 5,862 µg/L at the surface water POC. If they do exceed at the surface water POC, reconvene the planning team to evaluate and select a remedial action. Otherwise, continue monitoring iron concentrations in surface water.

The decision statements were expanded to compile rules for data use that reflect various combinations of contaminants, environmental media, and applicable criteria.

Groundwater Monitoring Decision Rules

Statistical trend analysis using the Mann-Kendall test is discussed in the Navy optimization guidance document (Battelle 2008) and was used in the *Five-Year Review, Operable Units 1, 4, 11, and 13*, (Tetra Tech August 2008), and in the *2011 Annual Groundwater Monitoring Report for Long-Term Monitoring, Operable Unit 1* (Tetra Tech, *in press*).

Decision Rule #3:

If a Mann-Kendall test at 10 percent significance level indicates that the concentration of any monitored parameter increases significantly in any groundwater monitoring well, the project team will convene to determine an appropriate course of action (e.g., increase frequency of monitoring or review groundwater geochemical data); otherwise, continue scheduled monitoring for that well. This rule was established to protect against the potential that parameter concentrations are increasing, regardless of whether the concentrations are greater than or less than the Performance Criteria. Even if parameter concentrations are less than the applicable criteria, an increasing trend could indicate that the concentrations will eventually become greater than the applicable criteria.



Decision Rules #4:

If any ROD parameter concentration including arsenic in any groundwater well is less than or equal to the Performance Criteria in groundwater (with the exception of compliance wells which are expected to remain less than the Performance Criteria) for two or more consecutive rounds of semi-annual (or less frequent) sampling, and the concentration trend is not increasing (as determined by a Mann-Kendall trend test at 10 percent significance), stop monitoring for that chemical in the well; otherwise, continue the scheduled monitoring for that parameter in the well. This rule was established to comply with the ROD.

Surface Water Monitoring Decision Rules

Decision Rule #5:

If a Mann-Kendall trend test at 10 percent significance indicates that the iron concentration trend is increasing at any monitored surface water location, the project team will convene to determine an appropriate course of action (e.g., increase frequency of monitoring at the SW POC and other points, review groundwater geochemistry data); otherwise, continue scheduled monitoring for that location. This rule was established to protect against the potential that iron concentrations are increasing and potentially will increase at the SW POC, regardless of whether the concentrations are greater than or less than the SWCTL. Even if iron concentrations are less than the applicable criterion, an increasing trend could indicate that the concentrations will eventually become greater than the criterion.

Decision Rule #6:

If the iron concentration at the SW POC immediately upgradient of Bayou Grande exceeds the background threshold of 5,862 µg/L for two consecutive semi-annual (or less frequent) sampling rounds, and the concentration trend is not increasing (as determined by a Mann-Kendall trend test at 10 percent significance), the Project Team will convene to evaluate and recommend a remedial action; otherwise, continue the scheduled monitoring at that location. This rule was established to comply with 62-780 F.A.C. and is designed to protect against a future unacceptable site condition with respect to iron concentrations.

Decision Rules #7:

If the iron concentration is less than the iron SWCTL at any surface water location for two or more consecutive semi-annual (or less frequent) rounds of sampling, optimize the monitoring program to reduce the frequency of monitoring for that chemical; otherwise, continue the



scheduled monitoring at that location. Optimization shall be conducted in accordance with current Navy groundwater optimization guidance to the extent that it is applicable to surface water. This rule was established to comply with 62-780 F.A.C.

The decision statements above were expanded to compile rules for data use that reflect various combinations of contaminants, environmental media, and applicable criteria. The number of rules was kept to a minimum while ensuring that in addition to identifying opportunities for monitoring system optimization, deteriorating groundwater or surface water conditions could be detected in time to implement an appropriate corrective action. Decision flow charts corresponding to these rules are presented on Figure 11-1, Decision Diagram for Use with Decision Rules.

If the contaminant source (i.e., the landfill) is removed, it will be necessary to establish new DQOs to accommodate this significant change in site configuration. The most important change may be to incorporate a decision rule that permits eventual discontinuation of all sampling and analyses.

11.6 Decision Performance Criteria

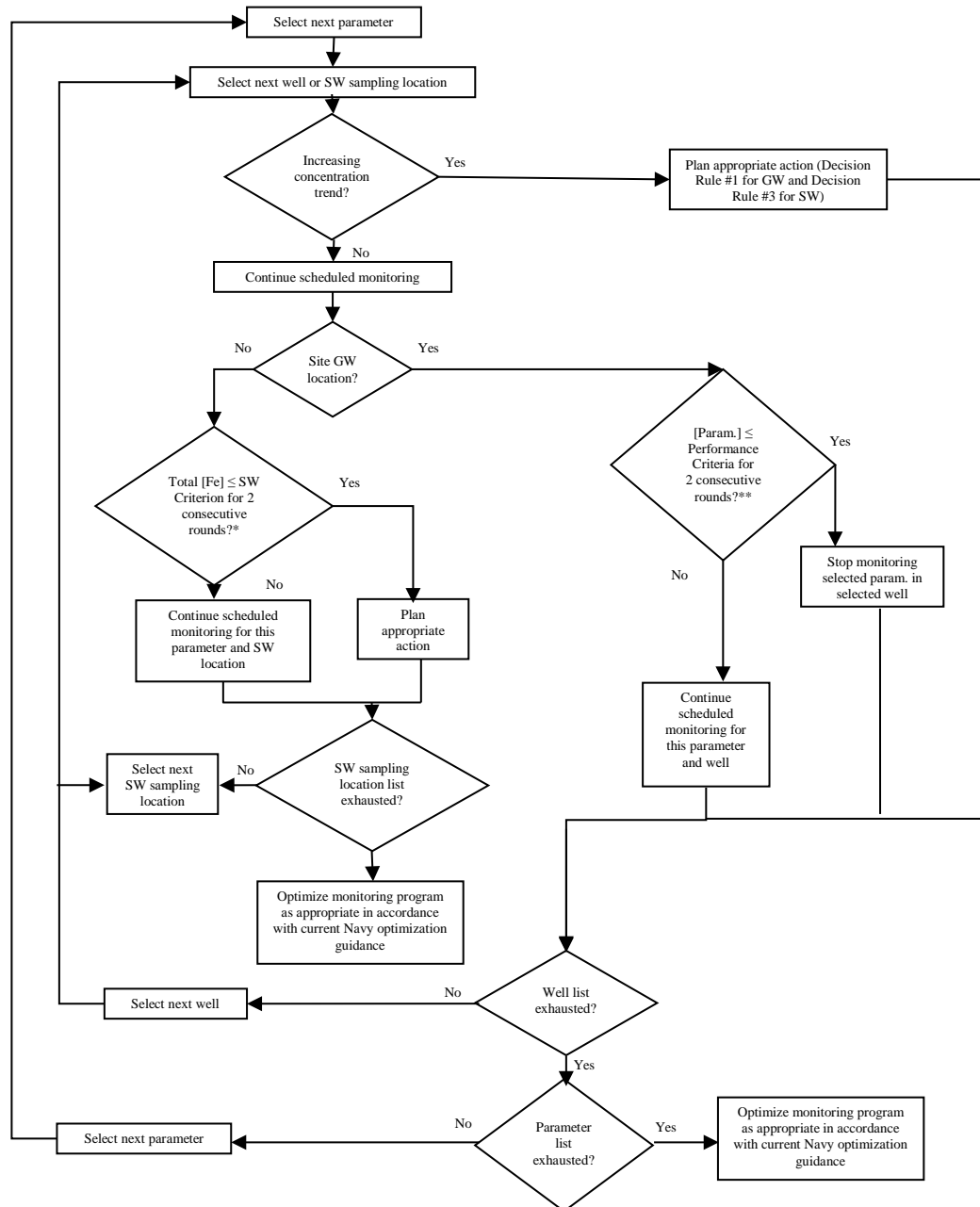
Individual measured concentrations in each monitoring well will be compared without adjustment to the Performance Criteria (ROD parameters including arsenic). No statistical criteria will be involved; however, data quality will be evaluated as part of the verification/validation and the data usability processes described in Worksheets #34 through #37. Failure to meet validation targets or limitations on data use identified during data usability assessment shall be described in the project report.

Initial Mann-Kendall trend detection shall be at the 10 percent significance level. At least four samples shall be collected in a particular well before this trend testing is implemented. Previously generated data and new data may be combined to construct trend graphs.

11.7 Optimize the Design for Obtaining Data

Details of the groundwater and surface water sampling design, rationale, and locations are provided on Worksheets #17 and #18. These details identify which groundwater monitoring wells and surface water sampling locations are to be sampled, the analyses to be conducted on each sample, and the reasons for sampling and analysis of each well and surface water sampling location.

Figure 11-1 Decision Diagram for Use with Decision Rules



* SW Criterion is 5,862 µg/L for iron at the point of compliance in Wetland 4D.
** GW criterion is the Performance Criteria Param = target parameter
SW = Surface Water
GW = Groundwater
Fe = Iron



SAP WORKSHEET #12: FIELD QUALITY CONTROL SAMPLES

(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table — Field QC Samples				
QC Sample	Analytical Group	Frequency	Data quality Indicators	Measurement Performance Criteria
Trip Blanks	VOCs	One per cooler containing VOC samples	Bias/Contamination	No analytes $\geq \frac{1}{2}$ LOQ, except common laboratory contaminants, which must be $<$ LOQ.
Equipment Rinsate Blank	VOCs and Metals	One per event per matrix per sampling equipment	Bias/Contamination	No analytes $\geq \frac{1}{2}$ LOQ, except common laboratory contaminants, which must be $<$ LOQ.
Field Duplicate	VOCs and Metals	One per 20 field samples	Precision	RPD must be ≤ 30 (VOCs) RPD must be ≤ 20 (Metals)
Matrix Spike/Matrix Spike Duplicate	VOCs and Metals	One pair per 20 field samples	Accuracy/Bias/ Precision	Percent recoveries — DoD QSM Limits RPD must be ≤ 30 (VOCs) RPD must be ≤ 20 (Metals)
Cooler Temperature Indicator	VOCs	One per cooler	Representativeness	Temperature must be above freezing and less than or equal to 6 °C.

Notes:

QC	=	Quality Control
VOC	=	Volatile organic compound
LOQ	=	Limit of quantitation
DoD QSM	=	<i>Department of Defense Quality Systems Manual for Environmental Laboratories</i> , Version 4.2, October 2010, or most recent version at the time of sampling
RPD	=	Relative percent difference



SAP WORKSHEET #13: SECONDARY DATA CRITERIA AND LIMITATIONS TABLE

(UFP-QAPP Manual Section 2.7)

Secondary Data Criteria and Limitations Table				
Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation/collection dates)	How Data Will Be Used	Limitations on Data Use
Record of Decision	EnSafe Inc., <i>Final Record of Decision, Operable Unit 1, Naval Air Station Pensacola, Pensacola, Florida</i> , September 25, 1998	<i>Originating Organization:</i> EnSafe Inc. <i>Data Types:</i> contaminant source areas, plume areas, source control and groundwater remedies, contaminants of concern, remedial goals, conceptual site model <i>Data Collection Dates:</i> 1993 — 1998	Contaminants of concern, remedial goals, and conceptual site model will be used as basis for long term monitoring.	None
Optimization Plan	Tetra Tech NUS, Inc., <i>Optimization Study Report for Operable Unit 1, Naval Air Station Pensacola, Pensacola, Florida</i> , (Rev.), April 2008	<i>Originating Organization:</i> Tetra Tech NUS, Inc. <i>Data Types:</i> Groundwater and Surface Water	Long Term Monitoring optimization	None
Technical Memorandum	Tetra Tech, <i>Technical Memorandum, Reconnaissance Phase Flow Control Pilot Study, Operable Unit 1, NAS Pensacola, Pensacola, Florida</i> , August 2009	<i>Originating Organization:</i> Tetra Tech NUS, Inc. <i>Data Types:</i> Surface Water	Established surface water point of compliance location; background iron criteria were calculated based on the historical site data.	None
Historical Analytical Data	Naval Installation Restoration Information Solution (NIRIS) database	<i>Originating Organization:</i> Prior U.S. Navy contractors <i>Data Types:</i> Location identifiers, coordinates, Geographic Information System maps, electronic analytical results <i>Data Collection Dates:</i> 1993 — 2012	Data obtained from NIRIS will be used to compare new long-term monitoring data to prior results to assess post-closure conditions.	None
Historical Analytical Data	Tetra Tech NUS, Inc., <i>1st through 5th Annual Monitoring Report for Long-Term Monitoring for Operable Unit 1, NAS Pensacola</i> , January 2001 through December 2006	<i>Originating Organization:</i> Tetra Tech NUS, Inc. <i>Data Types:</i> groundwater and surface water results <i>Data Collection Dates:</i> 2000 — 2005	Trend analysis, optimization, corrective action effectiveness, determinations, and ultimately, the cease of operations.	None
Historical Analytical Data	TetraTech, 2010 Annual Groundwater Monitoring Report for Long-Term Monitoring, Operable Unit 1, Naval Air Station Pensacola, Pensacola, Florida, 2012	<i>Originating Organization:</i> Tetra Tech NUS, Inc. <i>Data Types:</i> groundwater and surface water results <i>Data Collection Dates:</i> May and November 2010	Trend analysis, optimization, corrective action effectiveness, determinations, and ultimately, the cease of operations.	None
Historical Analytical Data	TetraTech, 2011 Annual Groundwater Monitoring Report for Long-Term Monitoring, Operable Unit 1, Naval Air Station Pensacola, Pensacola, Florida, <i>in press</i>	<i>Originating Organization:</i> Tetra Tech NUS, Inc. <i>Data Types:</i> groundwater and surface water results <i>Data Collection Dates:</i> August 2011 and January 2012	Trend analysis, optimization, corrective action effectiveness, determinations, and ultimately, the cease of operations.	None



SAP WORKSHEET #14: SUMMARY OF PROJECT TASKS

(UFP-QAPP Manual Section 2.8.1)

The following project tasks are summarized below:

- Field Tasks
- Analytical Tasks
- Data Management and Review
- Project Report

Field Tasks

The following describes field tasks and procedures that are applicable to this investigation. Semi-annual sampling is anticipated to take place every November and May until concentrations are reduced to below the applicable criteria. Field tasks will be performed in accordance with the Resolution Consultants and U.S. EPA standard operating procedures (SOPs) in Appendix B.

Mobilization/Demobilization — Mobilization will consist of the delivery, assembly (in satisfactory working order), and secure storage of necessary equipment, materials, and supplies, along with the acquisition of personnel and vehicle base access badges. The Resolution Consultants field team leader (FTL), or designee, will coordinate with the NAS Pensacola point of contact to identify appropriate locations for the temporary storage of equipment and supplies.

A field team orientation meeting will be conducted prior to starting the fieldwork to familiarize the team personnel with the site-specific health and safety requirements, the objectives and scope of the field activities, and chain-of-command. This meeting will be attended by the field staff and conducted by the FTL. A site walkover will be conducted prior to field activities and the FTL will identify related field support areas and requirements. Figure 10-5 shows the LTM sampling locations.

Demobilization will consist of the prompt and timely removal of equipment, materials, and supplies from the site, at the completion of fieldwork. Demobilization also includes the cleanup and removal of waste generated during the investigation.

Utility Clearance — Before beginning any intrusive activities, Resolution Consultants will coordinate utility clearance in accordance with Resolution Consultants SOP 3-01 with the Facility and Sunshine State One Call. The Facility and utility companies subscribed to Sunshine State One Call will



identify and mark utilities that may be present in the proposed well installation area. A NAS Pensacola excavation permit will be completed and submitted with the appropriate supporting documentation to the base a minimum of 10 days before well installation.

Monitoring Well Installation and Development — One shallow monitoring well (01UPGW2) will be installed at the proposed location shown on Figure 10-5. The well is intended to monitor background concentrations of chemicals in groundwater upgradient of OU1. The well will be installed to an approximate depth of 30 feet bgs with a 10-foot well screen bracketing the water table. The estimated depth is based on the ground surface and groundwater elevations in monitoring wells near the proposed location. The actual depth of the screened interval will be based on field observations of the lithology at this location. Soil lithology will be logged in accordance with Resolution Consultants SOP-3-16 and the Unified Soil Classification System.

The well will be installed using hollow-stem auger drilling methods in accordance with Resolution Consultants SOP-3-12. The monitoring well will be 2-inch nominal diameter, and will be constructed of Schedule 40 polyvinyl chloride screen and casing. The screen will be 0.010-inch slot size with 20 to 40 mesh size filter pack. The filter pack shall be set to a minimum of 1 foot above the top of screen and a minimum of 2-foot thick bentonite seal shall be installed above the sand pack. The remaining annular space from the top of the bentonite seal to the ground surface shall be grouted. The well will be completed with a stick-up protective guard pipe.

The monitoring well will be developed through overpumping in accordance with Resolution Consultants SOP-3-13 after installation, but not before a 24-hours set time from the final annular grouting.

Surveying — The horizontal location, elevation and top-of-riser elevation for the new monitoring well will be surveyed by a Florida registered professional land surveyor in accordance with Resolution Consultants SOP-3-07. The top-of-riser elevation will be surveyed to the nearest 0.01 foot and will be tied into the existing site datum (North American Vertical Datum 1988) and other monitoring wells at the site.

Field Calibration Procedures — Field equipment will be calibrated in accordance with the SOPs identified on Worksheet #21. Documentation of the field equipment calibration is required. Field equipment should be calibrated at the beginning and end of each day. Field instrument calibration measurement performance criteria are presented in Worksheet #22.



Groundwater and Surface Water Sampling

- Reference Worksheet #21 for the appropriate groundwater sampling SOPs.
- Complete all applicable forms (monitoring well sampling logs, Safe Work Assessment Permit) on a daily basis.
- Review health and safety plan with field sampling crews.
- Groundwater Level Measurements
 - Water level measurements will be conducted in all wells before the start of purging activities. Water level measurements will be recorded to the nearest 0.01 foot and referenced to a top of casing mark designated on each well. The water level indicator will be decontaminated before conducting measurements and immediately after each well.
- Collect Groundwater Samples:
 - All monitoring wells and piezometers will be purged prior to sampling using low-flow sampling techniques. Worksheets #17 and #18 specify the sample locations and target analytes for this investigation, and Worksheet #23 specifies the analytical methods to be used. After collection, the samples will be placed in a cooler, chilled with ice, and shipped under chain-of-custody protocol to the appropriate laboratories for analysis.
 - Field measurements of DO, ORP, turbidity, pH, temperature, and specific conductance will be made every 5 minutes. Once parameters are stable, water samples will be obtained using the same pump and flow rate. If the water-quality parameters do not stabilize, three to five well volumes will be purged from the well (unless the well is pumped dry) and then the well will be sampled using low-flow techniques.
 - The sample naming scheme will be in accordance with the established convention for this site. See Worksheet #18 for wells to be sampled semi-annually, designated sample identifications, and analyses.



- Monitoring well and piezometer locations are shown on Figure 10-5.
- Worksheet #19 specifies the required bottle types, preservation, target analytes, analytical methods, and holding times for this investigation.
- Collect Surface Water Samples:
 - Surface samples will be collected from three locations. The surface water sampling procedures provided in SESDPROC-201-R3 will be followed. SESDPROC-201-R3 establishes the procedure for standard grab surface water sampling. Surface grab samples will be collected from the top 12 inches of the water column above the sediment most likely to be impacted by inflow of surface runoff or drainage.
 - The sample naming scheme will be in accordance with the established convention for this site. See Worksheet #18 for locations to be sampled semi-annually, designated sample identifications, and analyses.
 - Surface water sampling locations are shown on Figure 10-5.
 - Worksheet #19 specifies the required bottle types, preservation, target analytes, analytical methods, and holding times for this investigation.

Field Quality Control (QC) Tasks — Field QC samples will be collected as part of each sample round, including field duplicates, trip blanks, and equipment rinsate blanks. In addition, extra sample volume will be collected for the laboratory QC analysis of matrix spike and matrix spike duplicate analysis. Worksheet #20 presents the field QC sample summary.

Decontamination — Decontamination of sampling equipment will not be necessary for dedicated and disposable sampling equipment. Decontamination of reusable sampling equipment (e.g., water level indicators) will be conducted prior to sampling and between samples at each location. Decontamination of major equipment and sampling equipment will be in general accordance with Resolution Consultants SOP-3-06.



Investigation-Derived Waste (IDW) — IDW includes excess environmental media, decontamination fluid, used personal protective equipment (PPE), used disposable sampling equipment, etc. IDW generated during the sampling activities will be managed in an environmentally responsible manner consistent with NAS Pensacola requirements (e.g., designation of staging areas) and SOP-3-05. The objectives of the IDW management are:

- Management of IDW in a manner that prevents contamination of uncontaminated areas (by IDW) and that is protective of human health and the environment.
- Minimization of IDW, thereby reducing costs and the potential for human or ecological exposure to contaminated materials.
- Managed, characterized, and disposed in compliance with federal and state requirements that are applicable or relevant and appropriate requirements.

Disposable PPE and disposable sampling equipment/materials will be disposed of as general refuse at an approved disposal location.

Analytical Laboratory Tasks

Chemical analysis will take place semi-annually, coinciding with the November and May sampling events. Data package turnaround time is anticipated to be 21 days from sample receipt.

Chemical analysis for VOCs, metals, and geochemistry will be performed by Empirical Laboratories, LLC (Empirical), who is currently certified by Florida Department of Health (FDOH) National Environmental Laboratory Accreditation Program (NELAP) and Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) for these analyses. Pace Analytical Energy Services LLC (formerly Microseeps Inc.), will perform hydrogen and carbon dioxide analysis and they are certified by FDOH NELAP. Certificates for both laboratories may be found in Appendix C and each laboratory's accreditation will be verified prior to each sampling event. Analyses will be performed in accordance with the analytical methods specified in Worksheet #19. Empirical will strive to meet the project action levels as shown in Worksheet #15 and will perform chemical analyses following laboratory-specific SOPs cited on Worksheet #23.



Data Management

Data management will take place semi-annually, coinciding with the November and May sampling events and after receipt of the laboratory data packages, which are anticipated 21 days from sample receipt.

The following describes data management procedures that are applicable to the LTM effort.

Data Handling and Management — After each LTM event is completed, the field sampling log sheets will be organized by date and media and filed in the project files. The field logbooks for this project will be used only for this site, and will also be categorized and maintained in the project files after the completion of the field program.

Data Handling and Management — The principal data generated for this project will be from field data and laboratory analytical data. The field forms, chain of custody, air bills, and logbooks will be placed in the project files after the completion of the field program. The field logbooks for this project will be used only for this site, and will also be categorized and maintained in the project files after the completion of the field program. All project records will be maintained in a secure location. Laboratory data, provided in electronic format, will be verified for accuracy prior to use and during the data validation process. After data are validated, the electronic data results will be uploaded into the Resolution Consultants database for use in data evaluation and subsequent report preparation. The project database will be located on a secure network, which is password protected. The Resolution Consultants TOM (or designee) is responsible for the overall tracking and control of data generated for the project.

Analytical Data Review and Validation — After receipt of analytical laboratory results, Resolution Consultants will verify data completeness as specified on Worksheet #34. To ensure that the analytical results meet the project quality objectives, the laboratory data will undergo verification and validation, without raw data review, as cited in Worksheets #34 through #36 and described below. The usability assessment processes are described in Worksheet #37.

Prior to data validation, electronic laboratory data will be verified for accuracy against the hardcopy laboratory report and the electronic quality assurance project plan (eQAPP) will be established using the project-specific criteria defined in Worksheets #12, #19, and #28. The laboratory will be requested to resubmit electronic data found to be inaccurate.



During the data validation process, the Resolutions Consultant's Data Validation Assistant (DVA) tool will be used to review method accuracy and precision data from field and laboratory QC samples contained in the laboratory electronic data deliverable (EDD) and qualify that data according to the project-specific eQAPP. The DVA tool uses the power of EarthSoft's EQuIS relational database to assemble a series of Excel worksheets into a DVA workbook for the validator that present:

- data validation QC elements that they need to review, compared to control limits stored in the project-specific eQAPP.
- associated sample results for duplicated samples and blanks.
- a place to make the necessary qualifications and result updates directly into an electronic format documentation of qualifications using coded reasons.
- list all samples affected by the qualification.

Laboratory calibration will be assessed against the criteria presented in Worksheet #24 using the hardcopy laboratory report and the results of these findings will be added to the Excel DVA workbook. The DVA workbook ultimately serves as an EDD to update the project database with the validator's changes. Using standard EQuIS tools that check and load data, qualifiers and edits are directly uploaded to the database thereby eliminating the manual data entry process and allowing for 100% of data to be reviewed prior to uploading to the project database.

Data Tracking and Control — The TOM (or designee) is responsible for the overall tracking and control of data generated for the project. Data are tracked from its generation to its archiving in the project specific files. The project chemist (or designee) is responsible for tracking the samples collected and shipped to the contracted laboratory. Upon receipt of the data packages from the analytical laboratory, the project chemist will oversee the data validation effort, which includes verifying that the data packages are complete and that results for all samples have been delivered by the analytical laboratory.

Resolution Consultants shall submit all Administrative Record Files, Site Files, and Post Decision Files in accordance with the specifications defined in the NAVFAC *Environmental Restoration Recordkeeping Manual* (NAVFAC 2009). Additionally, Resolution Consultants will update and



manage the project related documents, data, and maps in NIRIS. Project related spatial data including maps, models, and associated collected or created data will also be uploaded into NIRIS. All documentation submittals for NIRIS will be coordinated with the RPM.

Data Storage, Archiving, and Retrieval — After the data are validated, the data packages are entered into the Resolution Consultants' Navy CLEAN file system and archived in secure files. The field records including field logbooks, sample logs, chain-of-custody records, and field calibration logs will be submitted by the Resolution Consultants FTL to be entered into the Navy CLEAN file system before archiving in secure project files. Project files are audited for accuracy and completeness. Project files will be kept in a secured, limited access area and at the completion of the Navy contract. Files will be shipped to the Federal Records Center (FRC) for storage where the files will remain until 50 years after the last decision document for NAS Pensacola. Final validated environmental data will be uploaded into the NIRIS database.

Project Reports

Project documents and records obtained during the course of project activities (e.g., calibration logs, health and safety certificates, etc.) are identified on Worksheet #29.

LTM Reports will be issued annually after two semi-annual sampling events. The reports will include appropriate sections concerning site background, investigation activities, physical characteristics, nature and extent of contamination, trend analysis, and conclusions and recommendations.

Each LTM Report will be issued in draft to NAVFAC SE for initial review. NAVFAC SE comments will be addressed and the draft final report will be issued for regulatory review. Resolution Consultants will prepare responses to comments from U.S. EPA and FDEP reviewers.



SAP WORKSHEET #15: REFERENCE LIMITS AND EVALUATION TABLES

(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table							
Matrix:	Groundwater (Monitoring Wells)						
Analytical Group:	Volatile Organic Compounds						
Analyte	CAS No.	Project Action Level (µg/L) ¹	Project Action Level Source (µg/L)	Project Quantitation Limit Goal (µg/L)	Laboratory Limit of Quantitation ² (µg/L)	Laboratory Limit of Detection ² (µg/L)	Laboratory Method Detection Limit ² (µg/L)
Benzene	71-43-2	1	FDEP GCTL	0.33	1	0.5	0.25
Chlorobenzene	108-90-7	100	FDEP GCTL/ U.S. EPA MCL	33	1	0.5	0.25
Vinyl chloride	75-01-4	1	FDEP GCTL	0.33	1	0.5	0.25

Notes:

¹ Project Action Level references: FDEP GCTLs at http://www.dep.state.fl.us/waste/quick_topics/rules/documents/62-777/62-777_TableI_GroundwaterCTLs.pdf.

² Laboratory quantitation, detection, and method detection limits are provided by Empirical Laboratories, LLC and may vary during the course of long-term monitoring.

FDEP = Florida Department of Environmental Protection

PQL = Practical quantitation limit

µg/L = Micrograms per liter

GCTL = Groundwater Cleanup Target Level

U.S. EPA MCL = United States Environmental Protection Agency Maximum Contaminant Level



SAP WORKSHEET #15: REFERENCE LIMITS AND EVALUATION TABLES (continued)

Reference Limits and Evaluation Table							
Matrix:	Groundwater (Monitoring Wells)						
Analytical Group:	Metals						
Analyte	CAS No.	Project Action Level (µg/L) ¹	Project Action Level Source (µg/L)	Project Quantitation Limit Goal (µg/L)	Laboratory Limit of Quantitation ² (µg/L)	Laboratory Limit of Detection ² (µg/L)	Laboratory Method Detection Limit ² (µg/L)
Nickel	7440-02-0	100	FDEP GCTL	33	2.5	1.5	0.75
Arsenic, Total	7440-38-2	10	FDEP GCTL/ U.S. EPA MCL	3.4	2.5	1.5	0.75

Notes:

¹ Project Action Level references: Reference concentrations are from the *Site 1 Remedial Investigation Report* (EnSafe/Allen & Hoshall, 1996), groundwater cleanup target levels (FDEP GCTLs) at http://www.dep.state.fl.us/waste/quick_topics/rules/documents/62-777/62-777_TableI_GroundwaterCTLs.pdf, and maximum contaminant levels (MCLs) at <http://water.epa.gov/drink/contaminants/index.cfm#List>.

² Laboratory quantitation, detection, and method detection limits are provided by Empirical Laboratories, LLC and may vary during the course of long-term monitoring.

FDEP = Florida Department of Environmental Protection
 PQL = Practical quantitation limit
 µg/L = Micrograms per liter
 GCTL = Groundwater Cleanup Target Level
 U.S. EPA MCL = United States Environmental Protection Agency Maximum Contaminant Level



SAP WORKSHEET #15: REFERENCE LIMITS AND EVALUATION TABLES (continued)

Reference Limits and Evaluation Table							
Matrix:	Groundwater (Piezometers) and Surface Water						
Analytical Group:	Total Iron						
Analyte	CAS No.	Project Action Level ¹ (µg/L)	Project Action Level Source (µg/L)	Project Quantitation Limit Goal (µg/L)	Laboratory Limit of Quantitation ² (µg/L)	Laboratory Limit of Detection ² (µg/L)	Laboratory Method Detection Limit ² (µg/L)
Iron	7439-89-6	2,360	Freshwater Background Threshold Value	1,900	25	15	7.5

Notes:

¹ Project Action Level references: Reference concentration is from the OU 16 (Site 41), NAS Pensacola Wetlands Remedial Investigation Report *Naval Air Station Pensacola, Pensacola Florida* (EnSafe 2005).

² Laboratory quantitation, detection, and method detection limits are provided by Empirical Laboratories, LLC and may vary during the course of long-term monitoring.

µg/L = Microgram per liter



SAP WORKSHEET #15: REFERENCE LIMITS AND EVALUATION TABLES (continued)

Reference Limits and Evaluation Table							
Matrix:	Groundwater (Monitoring Wells)						
Analytical Group:	Geochemical Parameters						
Analyte	CAS No.	Project Action Level (mg/L)	Project Action Level Source (mg/L)	Project Quantitation Limit Goal (mg/L)	Laboratory Limit of Quantitation ¹ (mg/L)	Laboratory Limit of Detection ¹ (mg/L)	Laboratory Method Detection Limit ¹ (mg/L)
Alkalinity	471-34-1	—	—	—	1	1	1
Chloride	16887-00-6	—	—	—	0.5	0.33	0.17
Nitrate (as Nitrogen)	14797-55-8	—	—	—	0.25	0.1	0.033
Sulfate	14808-79-8	—	—	—	2.5	1.0	0.33
Methane	74-82-8	—	—	—	0.004	0.002	0.001
Ethane	74-84-0	—	—	—	0.004	0.002	0.001
Ethene	74-85-1	—	—	—	0.004	0.002	0.001
Total Organic Carbon	—	—	—	—	3	2.5	1.25
Hydrogen	1333-74-0	—	—	—	0.6 (nM)	0.074 (nM)	0.074 (nM)
Carbon dioxide	124-38-9	—	—	—	5.0	0.43	0.43

Notes:

¹ Laboratory quantitation, detection, and method detection limits are provided by Empirical Laboratories, LLC and may vary during the course of long-term monitoring.

mg/L = Milligrams per liter

— = Not applicable

nM = Nano Mole



SAP WORKSHEET #16: PROJECT SCHEDULE/TIMELINE TABLE

(UFP-QAPP Manual Section 2.8.2)

Year	Event	Begin Fieldwork	Laboratory Data Delivery	Draft Report	Draft Final Report	Final Report	Report Type
1	1 and 2	November 2012 and May 2014	December 2012 and June 2014	August 2014	March 2015	May 2015	Annual Groundwater Monitoring Report
2	3 and 4	November 2014 and May 2015	December 2014 and June 2015	August 2015	March 2016	May 2016	Annual Groundwater Monitoring Report
3	5 and 6	November 2015 and May 2016	December 2015 and June 2016	August 2016	March 2017	May 2016	Annual Groundwater Monitoring Report
4	7 and 8	November 2016 and May 2017	December 2016 and June 2017	August 2017	March 2017	May 2018	Annual Groundwater Monitoring Report



SAP WORKSHEET #17: SAMPLING DESIGN AND RATIONALE

(UFP-QAPP Manual Section 3.1.1)

Groundwater and surface water are the media of concern. Select VOCs and select total metals are the COCs for groundwater and total iron is the COC for surface water. Low to moderate concentrations are anticipated based on historical data. The LTM and operation and maintenance requirements for the sampling activities, including the following:

- 1) Locations and number of monitoring points
- 2) Frequency and duration of monitoring
- 3) Analyte list and QA/QC samples
- 4) Data evaluation, management, and reporting procedures

Worksheet #21 provides a list of SOPs that will be used during this LTM. Wells to be sampled semi-annually, designated sample IDs, and analyses are summarized on Worksheet #18. Worksheet #19 specifies the required bottle types, preservation, target analytes, analytical methods, and holding times.

An overview of the sampling strategies and rationales is provided below.

17.1 Groundwater Sampling Design, Locations, and Rationale

The groundwater sampling plan is a biased design that takes advantage of existing wells to represent various locations that are contaminated or could be contaminated and, therefore, represent potential unacceptable levels of risk. The main segments of the groundwater typically of interest are:

- Upgradient from the landfill (background). This area represents groundwater conditions as close as possible to site groundwater across OU 1, but without an impact from the landfill.
- The source area (i.e., the landfill and immediate downgradient edge of the landfill).
- Downgradient of the contaminant plume in as yet uncontaminated areas.
- The leading edge of the contaminant plume.
- Groundwater point of compliance (GW POC) wells that monitor the edge of the property prior to entering Bayou Grande to ensure that contamination migrating off-site does not exceed the performance criteria.



The existing shallow and intermediate wells, as shown on Figure 10-5, were chosen for sampling based on the local hydrogeology as described in Section 10.2. Specifically, existing shallow and intermediate wells were chosen in the path of likely groundwater contamination. Further details regarding existing wells including well screen depth and depth to groundwater can be reviewed within the most recent monitoring event, 2011 Annual Groundwater Monitoring Report for Long-Term Monitoring, Operable Unit 1 (Tetra Tech, *in press*).

There is no center line of contamination because contaminant or potential contaminant flow is radial from the landfill in northwestern to eastern directions. Samples collected from the well nest (01GS73 and 01GI74) located near Bayou Grande will provide a better understanding of groundwater upwelling and its influence on groundwater flow direction, and to ensure that groundwater concentrations do not exceed the applicable criteria at the GW POC. The other wells will be used to represent the portions of the plume identified above.

The existing background monitoring well (01UPGW1) has not been sampled since May 2010 because the well has been dry. A new background monitoring well, 01UPGW2, will be installed at the location shown on Figure 10-5 to facilitate continued monitoring of background concentrations.

Following installation and development of the new upgradient monitoring well, groundwater samples will be collected from selected wells to assess the current groundwater conditions. Seventeen existing monitoring wells, (plus one duplicate shallow well sample and one intermediate well sample for QC purposes) will be sampled semi-annually. The well locations that will be sampled are shown on Figure 10-5. The following groundwater monitoring wells will be sampled and analyzed for select VOCs, select metals, and geochemical parameters¹:

01UPGW2	01GGM33	01GS64	01GI32
01GI36	01GI41	01GI43	01GI44
01GI46	01GI59	01GI65	
01GS57	01GS62	01GS71	
01GS73	01GI74	01GI72	

¹ Select VOCs include benzene, chlorobenzene, and vinyl chloride. Select metals include nickel and arsenic. Select geochemical parameters include methane, ethane, ethene, nitrate, sulfate, chloride, total organic carbon, alkalinity, hydrogen and carbon dioxide.



Before collection of groundwater samples, static water levels will be measured at all wells. The wells will then be purged using a peristaltic pump. Purging will be conducted using a low-flow quiescent technique in accordance with EPA SESDPROC-301-R3, Groundwater Sampling.

Field parameters measured during the groundwater sampling activities will include pH, specific conductance, temperature, turbidity, DO, and ORP.

Piezometer Sampling Design, Locations, and Rationale

Piezometers at this site are intended to focus on the direction and magnitude of iron concentrations in groundwater discharging to Wetland 3 and the potential for discharge to Wetland 4. Groundwater samples will be collected from eight selected piezometers (four from each side of the interceptor trench) to assess the current iron concentrations and the passive effectiveness of the interceptor trench. The locations that will be sampled are shown on Figure 10-5. The following groundwater piezometer locations (plus one duplicate upgradient sample for QC purposes) will be sampled and analyzed for total iron only:

01PZ01	01PZ06	01PZ06A	01PZ07
01PZ07A	01PZ07B	01PZ08	01PZ10

Before collection of groundwater samples, static water levels will be measured at the piezometers. The piezometers will then be purged using a peristaltic pump. Purging will be conducted using a low-flow quiescent technique. Purging completion will be determined in accordance with EPA SESDPROC-301-R3. Groundwater samples will be collected in accordance with EPA SESDPROC-301-R3.

17.2 Surface Water Sampling Design, Locations, and Rationale

The surface water sampling strategy is a biased design to protect Bayou Grande from an influx of iron contamination from the landfill. This plan incorporates monitoring at the SW POC upgradient of Bayou Grande, as well as locations further upgradient of the SW POC. The upgradient sampling locations will provide an early warning if iron concentrations appear to be increasing toward unacceptable concentrations at the SW POC. Collection of background data is also planned so that it can be incorporated into future decision-making.



Surface water sample locations have been selected to provide representative coverage of Wetland 3 and Wetland 4D, which are located between the landfill and Bayou Grande. Surface water samples are intended to identify the fate as iron is transported through the Wetland 3 complex, beginning with the discrete spring discharge at the headwaters. Additional surface water sampling locations and objectives will be provided in the proposed OU16-OU1-OU2 Wetland SAP which is currently under development. The three surface water samples (plus one duplicate sample for QC purposes) will be collected from these locations and analyzed for total iron. The locations that will be sampled are shown on Figure 10-5. The following surface water locations will be sampled and analyzed for total iron:

01SW01

01SW02

01SW03

Surface water samples will be collected by directly filling the bottles from the surface water in accordance with EPA SESDPROC-201-R3. Water quality parameters (pH, specific conductance, turbidity, temperature, and DO) will be recorded for each sampling location.

17.3 Monitoring Program Optimization

Trend analysis will be conducted in accordance with the decision rules listed in Section 11.5.

During monitoring optimization, some of the steps that may be needed during the annual review of the monitoring program include:

- Review all analytical data generated during the last year. Determine whether any significant changes have occurred to contaminant concentrations or plume size and shape.
- Review available natural attenuation data, such as DO and total organic carbon (TOC) to confirm that conditions are still suitable for this process to occur. Review hydrogeologic data collected during the past year to determine whether the groundwater levels or flow directions have changed significantly.
- Determine whether contaminant concentrations are less than, or greater than, the applicable Performance Criteria (ROD parameters including arsenic), or SWCTLs (iron only).
- Verify that the receptors and exposure pathways identified in the CSM have not changed. If they have changed, update the CSM and other data quality objectives (DQOs) as necessary.



- Verify whether the Performance Criteria or SWCTLs have changed and modify the sampling strategy to incorporate changed values as appropriate — if changes are made, incorporate these changes into the ROD.
- Revise the CSM if it has changed significantly, and update it annually.

Plot the current plume location and make changes in the wells to be sampled and parameters to be analyzed in samples collected from the wells. The revised strategy must be consistent with current project DQOs.



SAP WORKSHEET #18: LOCATION-SPECIFIC SAMPLING METHODS/SOP REQUIREMENTS TABLE

(UFP-QAPP Manual Section 3.1.1)

Location-Specific Sampling Methods/SOP Table										
Matrix	Monitoring Well ID	Sample ID {Well ID-MMY}	Northing	Easting	Semi-Annual	Select VOCs ¹	Select Metals ²	Iron Only	Select Geochemical ³	Sampling SOP Reference
Groundwater	01GS64	01GS64-xxxx	506601.1563	1088513.375	Y	1	1		1	SESDPROC-301-R3
Groundwater	01GGM33	01GGM33-xxxx	1087666.75	507314.1563	Y	1	1		1	SESDPROC-301-R3
Groundwater	01GI32	01GI32-xxxx	507832.938	1089005.125	Y	1	1		1	SESDPROC-301-R3
Groundwater	01GI36	01GI36-xxxx	506214.75	1089187.25	Y	1	1		1	SESDPROC-301-R3
Groundwater	01GI41	01GI41-xxxx	506438.656	1087406.375	Y	1	1		1	SESDPROC-301-R3
Groundwater	01GI43	01GI43-xxxx	506507.562	1086440.25	Y	1	1		1	SESDPROC-301-R3
Groundwater	01GI44	01GI44-xxxx	507021.438	1086530.375	Y	1	1		1	SESDPROC-301-R3
Groundwater	01GI46	01GI46-xxxx	207907.094	1087495.75	Y	1	1		1	SESDPROC-301-R3
Groundwater	01GI59	01GI59-xxxx	507312.656	1087578.625	Y	1	1		1	SESDPROC-301-R3
Groundwater	01GI65	01GI65-xxxx	506513.375	1088578.25	Y	1	1		1	SESDPROC-301-R3
Groundwater	01UPGW2	01UPGW2-xxxx	TBD ⁴	TBD ⁴	Y	1	1		1	SESDPROC-301-R3
Groundwater	01GS62	01GS62-xxxx	505857.4007	1087739.176	Y	1	1		1	SESDPROC-301-R3
Groundwater	01GS57	01GS57-xxxx	507938.75	1089544.82	Y	1	1		1	SESDPROC-301-R3
Groundwater	01GS71	01GS71-xxxx	507680.3173	1090255.839	Y	1	1		1	SESDPROC-301-R3
Groundwater	01GI72	01GI72-xxxx	507679.7244	1090265.06	Y	1	1		1	SESDPROC-301-R3
Groundwater	01GS73	01GS73-xxxx	507983.87	1087534.07	Y	1	1		1	SESDPROC-301-R3
Groundwater	01GI74	01GI74-xxxx	507979.1	1087539.77	Y	1	1		1	SESDPROC-301-R3
Groundwater	01PZ01	01PZ01-xxxx	506306.696	1088601.602	Y			1		SESDPROC-301-R3
Groundwater	01PZ06	01PZ06-xxxx	506344.9789	1088593.392	Y			1		SESDPROC-301-R3
Groundwater	01PZ06	01PZ06A-xxxx	506344.9789	1088593.392	Y			1		SESDPROC-301-R3
Groundwater	01PZ07	01PZ07-xxxx	506267.0135	1088620.941	Y			1		SESDPROC-301-R3
Groundwater	01PZ07	01PZ07A-xxxx	506267.0135	1088620.941	Y			1		SESDPROC-301-R3
Groundwater	01PZ07	01PZ07B-xxxx	506267.0135	1088620.941	Y			1		SESDPROC-301-R3
Groundwater	01PZ08	01PZ08-xxxx	505968.5398	1088810.782	Y			1		SESDPROC-301-R3
Groundwater	01PZ10	01PZ10-xxxx	505991.8413	1088758.687	Y			1		SESDPROC-301-R3
Surface Water	SW01	SW01-xxxx	505996.477	1088725.6	Y			1		SESDPROC-201-R3
Surface Water	SW02	SW02-xxxx	506894.4034	1089198.701	Y			1		SESDPROC-201-R3
Surface Water	SW03	01SW03-xxxx	506566.6079	1088767.945	Y			1		SESDPROC-201-R3



Location-Specific Sampling Methods/SOP Table										
Matrix	Monitoring Well ID	Sample ID {Well ID-MMY}	Northing	Easting	Semi-Annual	Select VOCs ¹	Select Metals ²	Iron Only	Select Geochemical ³	Sampling SOP Reference
Field Quality Control Samples										
Groundwater Field Duplicate	01GI41	01GI41-xxxx	506438.656	1087406.375	Y	1	1			SESDPROC-301-R3
Groundwater Field Duplicate	01GS62	01GS62-xxxx	505857.4007	1087739.176	Y	1	1			SESDPROC-301-R3
Groundwater Field Duplicate	01PZ01	01PZ01-xxxx	506306.696	1088601.602	Y			1		SESDPROC-301-R3
Surface Water Field Duplicate	SW01	SW01-xxxx	505996.477	1088725.6	Y			1		SESDPROC-201-R3
Matrix Spike/ Matrix Spike Duplicate	TBD ⁵	TBD ⁵	TBD ⁵	TBD ⁵	Y	2	1	1		SESDPROC-301-R3, SESDPROC-201-R3
Equipment Blank	EBMMDDYY	NA	NA		Y	1	1			SESD-PROC-011-R4
Trip Blank	TBMMDDYY	NA	NA		Y	4				SESD-PROC-011-R4

Notes:

¹ Select VOCs include benzene, chlorobenzene and vinyl chloride.

² Select metals include nickel and arsenic.

³ Select geochemical parameters include methane, ethane, ethene, nitrate, sulfate, chloride, total organic carbon, alkalinity, hydrogen and carbon dioxide.

⁴ To be determined — coordinates of new monitoring well will be determine following installation.

⁵ To be determined — matrix spike/matrix spike duplicate samples will be determined in the field.

SOP = Standard operating procedure

VOCs = Volatile organic compounds

Y = Yes

xxxx = month (MM), year (YY), (e.g. 0213 for February 2013.)

NA = not applicable

Semi-annual sampling is anticipated to take place each November and May. For Field QC sample scoping, each sampling event is expected to last 4 days.

QA/QC sample collection frequency limited to VOCs and metals analysis:

Field Duplicate = One per 10 field samples

Equipment Blank = One per event

Trip Blank = One per cooler to the laboratory containing volatiles

MS/MSD = One pair per 20 field samples (including field QC samples); samples will be identified in the field and collected in triplicate volume.



SAP WORKSHEET #19: FIELD SAMPLING REQUIREMENTS TABLE

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Analytical and Preparation Method/ Laboratory SOP Reference	Containers (number, size, and type)	Sample Volume	Preservation Requirements ⁽¹⁾	Maximum Holding Time ⁽²⁾ (preparation/analysis)
Groundwater	VOCs	SW-846 5030/8260B/ Empirical SOP 202	(3) 40 mL glass volatile vials	40 mL	hydrochloric acid to a pH less than 2; Cool to 0-6°C; no headspace	14 days
Groundwater	Metals	SW-846 3005A/6010C Empirical SOP 105	(1) 250 mL plastic	50 mL	nitric acid to a pH less than 2	180 days
Groundwater	Alkalinity	SM 2320B/ Empirical SOP 154	(1) 250 mL plastic	50 mL	Cool to 0-6 °C	28 days
Groundwater	Chloride, Sulfate	E300.0/ Empirical SOP 145	(1) 250 mL plastic	100 mL	Cool to 0-6 °C	28 days
Groundwater	Methane, Ethane, Ethene	RSK-175/ Empirical SOP 236	(3) 40 mL glass volatile vials	40 mL	hydrochloric acid to a pH less than 2; Cool to 0-6°C; no headspace	14 days
Groundwater	Total Organic Carbon	SW-846 9060A/ Empirical SOP 221	(1) 250 mL plastic	5 mL	hydrochloric acid or sulfuric acid to pH<2; Cool to 0-6 °C	28 days
Groundwater	Hydrogen, Carbon Dioxide	Pace (Microseeps) SOP AM20GAX	20 mL Headspace vial, set to atmospheric pressure with UHP Nitrogen and a stopper type septa	15 mL	none	14 days

Notes:

¹ Chemical, temperature, and light protected

² Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted

SOP = Standard operating procedure

VOCs = Volatile organic compounds

UHP = Ultra High Purity

mL = Milliliter

°C = Degrees Celsius



SAP WORKSHEET #20: FIELD QUALITY CONTROL SAMPLE SUMMARY TABLE

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/MSDs	No. of Equipment Blanks	No. of VOC Trip Blanks	Total No. of Samples to Laboratory per Sampling Event
Groundwater	Select VOCs ¹	17	2	2	1	4	26
Groundwater	Select Metals ²	17	2	1	1	0	21
Groundwater	Geochemical Parameters ³	17	0	0	0	0	17
Groundwater (Piezometer)	Total Iron Only	8	1	1	0	0	10
Surface Water	Total Iron Only	3	1	1	0	0	5

Notes:

¹ Select VOCs include benzene, chlorobenzene and vinyl chloride.

² Select metals include nickel and arsenic.

³ Select geochemical parameters include methane, ethane, ethene, nitrate, sulfate, chloride, total organic carbon, alkalinity, hydrogen and carbon dioxide.

No. = Number

VOCs = Volatile organic compounds

MS = Matrix spike

MSD = Matrix spike duplicate

For Field QC sample scoping, each sampling event is expected to require 4 days.

QA/QC sample collection frequency limited to VOCs and metals analysis:

Field Duplicate = One per 10 field samples

Equipment Blank = One per event

Trip Blank = One per cooler to the laboratory containing volatiles

MS/MSD = One pair per 20 field samples (including field QC samples); samples will be identified in the field and collected in triplicate volume.



SAP WORKSHEET #21: PROJECT SAMPLING SOP REFERENCES TABLE

(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Modified for Project Work? (Yes/No)	Comments
SOP-3-01	Utility Clearance, June 2012	Resolution Consultants	No	
SOP-3-02	Logbooks, May 2012	Resolution Consultants	No	
SOP-3-03A	Sample Labeling and Chain of Custody, August 2012	Resolution Consultants	No	
SOP-3-04A	Sample Handling, Storage and Shipping Procedures of Low Level Environmental Samples, May 2012	Resolution Consultants	No	
SOP-3-05	Investigation Derived Waste Management, May 2012	Resolution Consultants	No	
SOP-3-07	Land Surveying, August 2012	Resolution Consultants	No	
SOP-3-12	Monitoring Well Installation, May 2012	Resolution Consultants	No	
SOP-3-13	Monitoring Well Development, June 2012	Resolution Consultants	No	
SOP-3-16	Soil and Rock Classification, August 2012	Resolution Consultants	No	
SESDPROC-005-R2	Sample and Evidence Management, 29 January 2013	EPA Region 4	No	
SESDPROC-010-R5	Logbooks, 30 May 2013	EPA Region 4	No	
SESDPROC-011-R4	Field Sampling Quality Control, 5 February 2013	EPA Region 4	No	
SESDPROC-012-R3	Field Sampling and Measurement Procedures and Procedure Validation, 30 August 2012	EPA Region 4	No	
SESDPROC-100-R3	Field pH Measurement, 29 January 2013	EPA Region 4	No	
SESDPROC-101-R5	Field Specific Conductance Measurement, 30 August 2012	EPA Region 4	No	
SESDPROC-102-R3	Field Temperature Measurement, 4 February 2011	EPA Region 4	No	
SESDPROC-103-R3	Field Turbidity Measurement, 29 January 2013	EPA Region 4	No	
SESDPROC-105-R2	Groundwater Level and Well Depth Measurement, 29 January 2013	EPA Region 4	No	
SESDPROC-106-R3	Field Measurement of Dissolved Oxygen, 8 January 2014	EPA Region 4	No	
SESDPROC-108-R4	Equipment Inventory and Management, 6 February 2013	EPA Region 4	No	
SESDPROC-110-R3	Global Positioning System, 20 April 2011	EPA Region 4	No	
SESDPROC-111-R3	In Situ Water Quality Monitoring, 19 July 2013	EPA Region 4	No	



Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Modified for Project Work? (Yes/No)	Comments
SESDPROC-113-R1	Field Measurement of ORP, 29 January 2013	EPA Region 4	No	
SESDPROC-202-R2	Management of Investigation Derived Waste, 15 October 2010	EPA Region 4	No	
SESDPROC-205-R2	Field Equipment Cleaning and Decontamination, 20 December 2011	EPA Region 4	No	
SESDPROC-209-R2	Packaging, Marking, Labeling and Shipping of Environmental and Waste Samples, 20 April 2011	EPA Region 4	No	
SESDPROC-301-R3	Groundwater Sampling, 6 March 2013	EPA Region 4	No	
SESDPROC-201-R3	Surface Water Sampling, 28 February 2013	EPA Region 4	No	
SOP-SM-9	Gas Stripping Cell Instructions, not dated	Pace (Microseeps, Inc.)	No	

Notes:

The Field Team Leader, William Honea, will be responsible for overseeing sampling and collection activities identified in this table. Onsite support facilities will be provided, as necessary, by NAS Pensacola Site Manager/Point of Contact, Greg Campbell.

SOP = Standard Operating Procedure
EPA = Environmental Protection Agency
ORP = Oxidation-Reduction Potential



SAP WORKSHEET #22: FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION TABLE

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference	Spare Parts (Kept in Carrying Case)
Peristaltic Pump GeoTech Series II (or similar)	Visual Inspection Field checks as per manufacturer	Daily Beginning of each day	Pump is variable speed and therefore, acceptance is based on pumps ability to pump at rate necessary to achieve stable flow from various wells.	Operator correction or replacement	Resolution Consultants FTL or designee	Manufacturer's Manual SESDPROC-108-R4 SESDPROC-205-R2 SESDPROC-301-R3	Battery Pack Power Cords Screws Nuts Washers
Water Level Indicator Solinst Model 101 (or similar)	Visual Inspection Field checks as per manufacturer	Once upon receiving from vendor Then Daily	0.01 foot accuracy	Operator correction or replacement	Resolution Consultants FTL or designee	Manufacturer's Manual SESDPROC-105-R2	Batteries
Multi-Parameter Water Quality Meter YSI 556 Series (or similar)	Visual Inspection Calibration/ Verification	Daily Beginning of each day	pH \pm 0.2 standard units Specific Conductance \pm 10% Dissolved oxygen \pm 0.2 mg/L Temperature \pm 4 °Celsius Oxygen reduction potential 10 mV	Operator correction or replacement	Resolution Consultants FTL or designee	Manufacturer's Manual, SESDPROC-100-R3, SESDPROC-101-R5, SESDPROC-106-R3, SESDPROC-102-R3, SESDPROC-113-R1	Batteries Field-replaceable DO, pH, and pH/ORP probes DO membranes Calibration solutions
Turbidity Meter LaMotte Model 2020e (or similar)	Visual Inspection Calibration/ Verification	Daily Beginning of each day	Nephelometric Turbidity Unit 10%	Operator correction or replacement	Resolution Consultants FTL or designee	Manufacturer's Manual, SESDPROC-103-R3	Batteries Sample Cells Calibration solutions
Global Positioning System Trimble Geo XT (or similar)	Positioning	Beginning and end of each day used	Accuracy: sub-meter horizontal dilution of precision < 3, number of satellites must be at least six	Wait for better signal, replace unit, or choose alternate location technique	Resolution Consultants FTL or designee	Manufacturer's Manual, SESDPROC-110-R3	Battery Pack Spare stylus and tether

Notes:

FTL = Field team leader
SOP = Standard operating procedure
% = Percent
mg/L = Milligrams per liter
mV = Millivolts



Field measurement equipment will be checked for operation in accordance with the manufacturer's specifications. This includes battery checks, routine replacement of membranes, and cleaning of conductivity electrodes. All equipment will be inspected for damage when first handed out and when returned from use. Equipment used to gather, generate, or measure environmental data will be calibrated within the frequency stipulated by the SOP and manufacturer's instructions in such a manner that accuracy and reproducibility of results are consistent. Prior to use, field-measuring equipment will be examined to certify that it is in operating condition. Field personnel will be responsible for inspecting equipment before use and they will follow the manufacturer's instructions for assembly, operation, and maintenance of field instruments and equipment. They will verify that the calibration requirements have been met for the instruments used and that all equipment is in proper working condition prior to use. The preventive maintenance of field equipment is described in detail in the associated manufacturer's equipment manuals. Records of equipment maintenance will be maintained in the field logbook.

Field personnel will follow the manufacturer's instructions for assembly, operation, and maintenance of field instruments and equipment. Field personnel will verify that the instrument calibration requirements have been met for the instruments used and that all equipment is in proper working condition prior to use. All field instruments will be inspected upon receipt and each day prior to use. Inspections of instruments prior to their use shall consist of a general examination of the probes, wires, and electrical systems (battery check), and calibration check. If a field instrument proves faulty, the equipment will be taken out-of-service until corrective action can be performed to return the unit to working order. If appropriate, a substitute unit will be delivered to the site to ensure that the integrity of the work is not compromised.

Calibration is the process by which the correlation between instrument response and actual value of a measured parameter is determined. Field instruments will be calibrated daily before use according to the manufacturers' specifications. Field personnel will document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements. Instruments may also be re-calibrated during the day if field personnel consider it necessary. Instrument calibration will be recorded in the field logbook or on project-specific calibration forms. Field instruments will be calibrated according to SOPs in Appendix D.



Whenever field measurements do not fall within acceptance limits, corrective action should be taken to bring the analysis back into control. The corrective action should include: (1) finding the cause of the problem, (2) correcting the problem, including replacing equipment, (3) demonstrating the problem has been corrected by reanalyzing appropriate laboratory reference samples, if necessary, and (4) repeating the analyses of any investigative samples that may have been affected by the control problem, if necessary. Any preventative or corrective maintenance completed will be documented in the field notes.



SAP WORKSHEET #23: ANALYTICAL SOP REFERENCES TABLE

(UFP-QAPP Manual Section 3.2.1)

Laboratory Name and Address: Empirical Laboratories, LLC, 621 Mainstream Drive, Suite 270, Nashville, Tennessee 37228

Laboratory Point of Contact/Project Manager: Sonya Gordon, sgordon@empirlabs.com, 615-345-1115

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM	Modified for Project Work? (Yes/No)
SOP 202	<i>GC/MS Volatiles by EPA Method 624 & SW846 Method 8260B including Appendix IX Compounds</i> , Revision 26, 08 January 2013	Definitive	VOCs Groundwater	Gas Chromatography/ Mass Spectrophotometer	None	No
SOP 100	<i>Metals Digestion/Preparation</i> , Revision 25, 19 December 2013	Definitive	Metals Groundwater, Surface Water	NA	None	No
SOP 105	<i>Metals by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) Technique References: SW-846, Method 6010B, December 1996; SW-846, Method 6010C, Revision 3 February 2007; U.S. EPA, Method 200.7, June 1991; Standard Methods 19th Edition 2340B; 1995 U.S. EPA CLP, ILM 04.1. See Addendum for U.S. EPA CLPILM 05.2</i> , Revision 20, 01 September 2013	Definitive	Metals Groundwater, Surface Water	Inductively Coupled Plasma Atomic Emission Spectrometry	None	No
SOP 154	<i>Total Alkalinity, Carbonate, Bicarbonate by Method US EPA 310.1, SM2320B (20th and 21st Edition)</i> , Revision 9, 10 April 2013	Definitive ¹	Alkalinity Groundwater	pH Meter/Titrator	None	No
SOP 145	<i>Determination of Inorganic Anions in water by ION Chromatograph using the Dionex dx-500 Ion Chromatograph with Hydroxide Eluent and Dionex AS18 Column, U.S. EPA Method 300.0/SW846 Method 9056</i> , Revision 10, 06 August 2013	Definitive ¹	Chloride, Sulfate Groundwater	Ion Chromatograph	None	No
SOP 236	<i>Methane, Ethane, Ethene in Aqueous Samples by Modified RSK-175 (Automated Headspace)</i> , Revision 5, 11 July 2013	Definitive ¹	Methane, Ethane, Ethene Groundwater	Gas Chromatography/ Electron Capture Detector	None	No
SOP 221	<i>Total Organic Carbon (TOC) by SM5310C, SW846 Method 9060/9060A and Lloyd Kahn Method "Determination of TOC in Sediment"</i> , Revision 15, 11 October 2013	Definitive ¹	Total Organic Carbon Groundwater	Total Organic Carbon Analyzer	None	No
SOP QS08	<i>Technical/ Operational Definitions Minimum Essential Quality Control elements, and Laboratory Calibration Procedures (Major & Support Equipment)</i> , Revision 04, 31 October 2011 (reviewed 10 April 2013)	NA	NA	NA	None	No
SOP QS10	<i>Laboratory Sample Receiving, Log In and Storage</i> , Revision 20, 03 September 2013	NA	NA	NA	None	No



Laboratory Name and Address: Empirical Laboratories, LLC, 621 Mainstream Drive, Suite 270, Nashville, Tennessee 37228

Laboratory Point of Contact/Project Manager: Sonya Gordon, sgordon@empirlabs.com, 615-345-1115

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM	Modified for Project Work? (Yes/No)
SOP QS11	Field Sampling & Bottle Kit Preparation, Revision 16, 27 January 2014	NA	NA	NA	None	No
SOP QS14	Analytical Laboratory Waste Disposal, Revision 8, 23 January 2013	NA	NA	NA	None	No
SOP 417	Traceability and Expiration Dates of Test-Related Chemicals for Organic and Inorganic Methods, Revision 6, 11 April 2013	NA	NA	NA	None	No
SOP 400	Laboratory Administrative Assistant SOP for Purchasing, Revision 8, 05 August 2013	NA	NA	NA	None	No

Notes:

¹ Analytical procedure produces definitive results; however, the end use of the data will be screening level to assess remedial effectiveness

SOP = Standard operating procedure

QSM = Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, October 2010

SOP = Standard operating procedure

VOCs = Volatile organic compounds

NA = Not applicable



SAP WORKSHEET #23: ANALYTICAL SOP REFERENCES TABLE (continued)

Laboratory Name and Address: Pace (Microseeps, Inc)., 220 William Pitt Way, Pittsburgh, Pennsylvania 15238

Laboratory Point of Contact/Project Manager: Robbin Robl, rrobl@microseeps.com, 412-826-5245

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM	Modified for Project Work? (Yes/No)
AM20GAx	<i>SOP for the Analysis of Biodegradation Indicator Gases, Revision 13, 5 September 2013</i>	Definitive ¹	Hydrogen and Carbon Dioxide	Gas Chromatography/ Flame Ionization Detector/Thermo Conductivity Detector/ Reduction Gas Detector	None	No
ADM06	<i>SOP for Regular Equipment Maintenance, Revision 5.0, 18 November 2011</i>	NA	NA	NA	None	No

Notes:

¹ Analytical procedure produces definitive results; however, the end use of the data will be screening level to assess remedial effectiveness

SOP = Standard operating procedure

QSM = Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, October 2010

SOP = Standard operating procedure



SAP WORKSHEET #24: ANALYTICAL INSTRUMENT CALIBRATION TABLE

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
GC/MS VOCs	Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples. Flagging criteria are not appropriate.	Analyst, Supervisor	Empirical SOP 202
GC/MS VOCs	ICAL Minimum five-point initial calibration for all analytes (six-points for quadratic)	Initial calibration prior to sample analysis	Average RF for SPCCs: ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane; and each analyte must meet <u>one</u> of the following: <u>Option 1</u> : %RSD for each analyte $\leq 15\%$; <u>Option 2</u> : linear least squares regression for each analyte: $r^2 \geq 0.99$; <u>Option 3</u> : non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Repeat calibration if criterion is not met	Analyst, Supervisor	Empirical SOP 202
GC/MS VOCs	Second source ICV	Once after each ICAL	All analytes within $\pm 20\%$ of expected value	Remake standard, recalibrate if necessary	Analyst, Supervisor	Empirical SOP 202
GC/MS VOCs	CCV	CV daily, before sample analysis, and every 12 hours of analysis time	RF for SPCCs: ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane; and %D ≤ 20 for all target compounds	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	Empirical SOP 202
GC/MS VOCs	RRT Evaluation	Prior to sample analysis	RRT of each target analyte within ± 0.06 RRT units of the RRT standard.	CCV fails, perform column maintenance, inspect pumps, and leak checks	Analyst, Supervisor	Empirical SOP 202
ICP-AES Metals	ICAL for all analytes: minimum one high standard and a calibration blank	Daily initial calibration prior to sample analysis	If more than one calibration standard is used, $r \geq 0.995$	Recalibrate and/or perform necessary equipment maintenance.	Analyst, Supervisor	Empirical SOP 105
ICP-AES Metals	Second source ICV	Once after each initial calibration	All analytes within $\pm 10\%$ of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification.	Analyst, Supervisor	Empirical SOP 105
ICP-AES Metals	CCV	CCV after every 10 samples and at the end of the analytical sequence	All analytes within $\pm 10\%$ of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification.	Analyst, Supervisor	Empirical SOP 105



Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
ICP-AES Metals	Low-level calibration check standard	Daily following calibration	All analytes within $\pm 20\%$ of expected value	Recalibrate and/or perform necessary equipment maintenance.	Analyst, Supervisor	Empirical SOP 105
ICP-AES Metals	Calibration blank	Once with each ICAL, after every 10 samples and at the end of an analytical sequence.	Determined concentration \leq LOD	Determine source of possible contamination, perform maintenance and recalibrate.	Analyst, Supervisor	Empirical SOP 105
ICP-AES Metals	Interference check solutions (ICS)	After beginning of the analytical run	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD ICS-AB: Within 20% of true value	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples. If corrective action fails, qualify all associated analyte results "Q."	Analyst, Supervisor	Empirical SOP 105
ICP-AES Metals	Linear Dynamic range/High level Check	Every 6 months and with major maintenance	90-110% recovery	Perform maintenance and/or reanalyze at lower concentration.	Analyst, Supervisor	Empirical SOP 105
Alkalinity	ICAL	Daily ICAL prior to sample analysis.	4 ± 0.05 pH units, 7 ± 0.05 pH units, 10 ± 0.10 pH units	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Supervisor	Empirical SOP 154
Alkalinity	CCV	Every 3 hours after calibration	7 ± 0.20 pH units for check	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Supervisor	Empirical SOP 154
Ion Chromatograph Chloride, Sulfate	ICAL — for all analytes: minimum three standards and a calibration blank	Daily initial calibration prior to sample analysis	$r \geq 0.995$	Correct problem, then repeat ICAL.	Analyst, Supervisor	Empirical SOP 145
Ion Chromatograph Chloride, Sulfate	ICV	Once after each ICAL, prior to beginning a sample run.	All project analytes within established retention time windows. Within $\pm 10\%$ of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Analyst, Supervisor	Empirical SOP 145



Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
Ion Chromatograph Chloride, Sulfate	CCV	After every 10 field samples and at the end of the analysis sequence.	All project analytes within established retention time windows. Within $\pm 10\%$ of true value.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Supervisor	Empirical SOP 145
GC/FID Methane, Ethane, Ethene	ICAL Minimum five-point initial calibration for all analytes (six-points for quadratic)	Upon instrument receipt, for major instrument changes, or when CCV does not meet criteria	Each analyte must meet <u>one</u> of the following: <u>Option 1</u> : %RSD for each analyte $\leq 20\%$; <u>Option 2</u> : linear least squares regression for each analyte: $r^2 \geq 0.99$; <u>Option 3</u> : non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Repeat calibration if criteria is not met	Analyst, Supervisor	Empirical SOP 236
GC/FID Methane, Ethane, Ethene	ICV	After Calibration	%D < 20%	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Supervisor	Empirical SOP 236
GC/FID Methane, Ethane, Ethene	CCV	Daily, and after every 10 field samples (not to exceed 12hrs), and at end of run	%D < 20%	If %D $\geq 20\%$ and sample result is ND, narrate. If %D $\leq 20\%$, reanalyze all samples since last successful CCV	Analyst, Supervisor	Empirical SOP 236
TOC Analyzer TOC	ICAL	Prior to sample analysis or instrument change, when instrument does not meet method criteria	Each analyte must meet <u>one</u> of the following: <u>Option 1</u> : linear least squares regression for each analyte: $r^2 \geq 0.99$; <u>Option 2</u> : non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Repeat calibration if criteria is not met	Analyst, Supervisor	Empirical SOP 221
TOC Analyzer TOC	ICV	After Calibration	%Ds must be: ICV $\pm 20\%$	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Supervisor	Empirical SOP 221
TOC Analyzer TOC	CCV	At the beginning and end of the sequence and every 10 field samples or every 5 samples if analyzing in quadruplicate	%Ds must be: CCV $\pm 20\%$ (water) CCV $\pm 30\%$ (soil)	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Supervisor	Empirical SOP 221



Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
GC/FID/ TCD/RGD Dissolved Gases	ICAL	Initial calibration prior to sample analysis. Re-analyze calibration following major instrument maintenance and/or failures of CCV	Coefficient of determination (r^2) is ≥ 0.995 ; linear or quadratic fit types.	Correct problem, then repeat ICAL.	Analyst, Supervisor	AMGax20
GC/FID/ TCD/RGD Dissolved Gases	ICV	Immediately following the initial calibration	All analytes within 15% of expected value; 85-115 percent recovery limits.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Supervisor	AMGax20
GC/FID/ TCD/RGD Dissolved Gases	CCV	Beginning and end of each analytical shift and every 15 analyzed field samples.	All analytes within 15% of expected value; 85-115 percent recovery limits.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Supervisor	AMGax20
GC/FID/ TCD/RGD Dissolved Gases	ICB/CCB	Follows each ICV/CCV	No analytes detected $> \frac{1}{2}$ LOQ.	Analyze ICB/CCB a second time. If the problem persists, re-analyze all samples since the last successful CCB.	Analyst, Supervisor	AMGax20

Notes:

SOP	=	Standard operating procedure	r^2	=	Least squares regression coefficient/coefficient of determination
GC/MS	=	Gas chromatograph/mass spectrometer	ICV	=	Initial calibration verification
VOCs	=	Volatile organic compounds	CCV	=	Continuing calibration verification
ICP-AES	=	Inductively coupled plasma-atomic emission spectrometry	CV	=	Calibration verification
GC/FID	=	Gas chromatography/flame ionization detector	RRT	=	Relative retention times
TOC	=	Total organic carbon	RGD	=	Reduction gas detector
TCD	=	Thermo conductivity detector	%D	=	Percent difference
ICAL	=	Initial calibration	r	=	Correlation coefficient
RF	=	Response factor	LOD	=	Limit of detection
SPCC	=	System performance check compound	ICS	=	Interference check solutions
%RSD	=	Percent relative standard deviation	LOQ	=	Limit of quantitation



SAP WORKSHEET #25: ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION TABLE

(UFP-QAPP Manual Section 3.2.3)

Laboratory preventive maintenance will be implemented in accordance with the Laboratory QA Plan and associated SOPs. At a minimum, all major instrumentation will have associated records and logbooks, including schedules and criteria for maintenance.

The laboratory's QA Manual and SOPs incorporate procedures for critical supplies and consumables, including standard supply sources, acceptance criteria, and for tracking and retrieving these materials. Each analytical SOP, in Appendix D, provides a comprehensive list and acceptance criteria of consumables such as solvents, reagents, buffer solutions, and other consumables required for each analytical method. Each laboratory analyst is responsible for checking and inspecting supplies and consumables for the task or analytical method they are assigned. Replacement consumable items will be obtained using the approved procedures and suppliers identified in the laboratory purchasing SOP. The following table identifies all analytical instrumentation that requires maintenance, testing, and inspection frequency, provides acceptance criteria, and corrective action requirements, and applicable laboratory SOP numbers.



Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Clean source, replace vacuum pump oil, replace filament, replace inlet liner, replace septa, and bake out instrument.	VOCs	Inspect vacuum pressure	Clean source and replace vacuum pump oil annually or as needed. Routine maintenance as necessary. Tune instrument after major maintenance. Inspect vacuum pressure daily.	BFB tune must meet the criteria listed in the method. Per instrument manufacturer's instructions	Inspect system and correct problem; Re-tune instrument if necessary. Perform new initial calibration.	Analyst, Department Manager	Empirical SOP 202
ICP-AES	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, and replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Metals	Torch, nebulizer chamber, pump, pump tubing	Prior to initial calibration and as necessary	Acceptable calibration or continuing calibration verification	Correct the problem and repeat calibration or continuing calibration verification	Analyst, Department Manager	Empirical SOP 105
pH Meter	Change buffer solutions or pH probe	Alkalinity	Change buffer solutions or pH probe	Before analysis begins, check every 3 hours	4 and 7 \pm .05 pH units, pH 10 \pm 0.10 pH units, \pm 0.20 pH units for check	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data	Analyst/ Supervisor	Empirical SOP 154
Ion Chromatograph	Check pump and conductivity detector	Chloride, Sulfate	Check pressure, flow, and conductivity.	Daily	Acceptable calibration criteria.	Repair and replace as needed.	Analyst, Department Manager	Empirical SOP 145
GC/FID	Replace septa, replace inlet liner, clip column, clean or replace FID jet, bake out detector, recondition column.	Methane, Ethane, Ethene	Check connections, replace disposables, bake out instrument, recondition column and perform leak checks.	Replace liner, septa, and clip column as indicated by instrument change in response and chromatography. If signal is suppressed, clean or replace FID jet. Bake out detectors and columns if signal elevated.	Per instrument manufacturer's instructions	Inspect system; correct problem; perform new initial calibration and affected samples.	Analyst, Department Manager	Empirical SOP 236



Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Total organic carbon Analyzer	Perform maintenance as needed.	Total organic carbon	Check waste container and gas cylinder.	Daily	Acceptable calibration criteria	Repair and replace as needed.	Analyst, Department Manager	Empirical SOP 221
GC/FID/TCD/RGD	Replace septa, replace cylinder, replace inlet liner, clip column, clean or replace FID jet, replace TCD filaments, change RGD activated carbon, bake out detector, recondition column.	Dissolved Gases	Check connections, replace disposables, bake out instrument, recondition column and perform leak checks.	Replace liner, septa, and clip column as indicated by instrument change in response and chromatography. If signal is suppressed, clean or replace FID jet, TCD filaments, and RGD activated carbon. Bake out detectors and columns if signal elevated.	Per instrument manufacturer's instructions	Inspect system; correct problem; perform new initial calibration and affected samples.	Analyst, Department Manager	Pace (Microseeps) SOP ADM06

Notes:

SOP = Standard operating procedure
 GC/MS = Gas chromatograph/mass spectrometer
 VOCs = Volatile organic compounds
 TCD = Thermo Conductivity Detector

BFB = Bromofluorobenzene
 ICP-AES = Inductively coupled plasma-atomic emission spectrometry
 GC/FID = Gas chromatography/flame ionization detector
 RGD = Reduction Gas Detector



SAP WORKSHEET #26: SAMPLE HANDLING SYSTEM

(UFP-QAPP Manual Appendix A)

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT	
Sample Collection (Personnel/Organization):	FTL/Resolution Consultants
Sample Packaging (Personnel/Organization):	FTL/Resolution Consultants
Coordination of Shipment (Personnel/Organization):	FTL/Resolution Consultants
Type of Shipment/Carrier:	Overnight via FedEx
SAMPLE RECEIPT AND ANALYSIS	
Sample Receipt (Personnel/Organization):	Sample Receiving Group/Empirical Laboratories, LLC and Pace (Microseeps Inc.)
Sample Custody and Storage (Personnel/Organization):	Sample Receiving Group/Empirical Laboratories, LLC and Pace (Microseeps Inc.)
Sample Preparation (Personnel/Organization):	Inorganic and Organic Preparation Personnel, Empirical Laboratories, LLC and Pace (Microseeps Inc.)
Sample Determinative Analysis (Personnel/Organization):	Inorganic and Organic Preparation Personnel, Empirical Laboratories, LLC and Pace (Microseeps Inc.)
SAMPLE ARCHIVING	
Field Sample Storage (No. of days from sample collection):	180 Days from Receipt of Samples
Sample Extract/Digestate Storage (No. of days from extraction/digestion):	180 Days from Receipt of Samples
SAMPLE DISPOSAL	
Personnel/Organization:	Waste Compliance Manager, Empirical Laboratories, LLC and Pace (Microseeps Inc.)
Number of Days from Analysis:	180 Days from Receipt of Samples

Note:

FTL = Field team leader



SAP WORKSHEET #27: SAMPLE CUSTODY REQUIREMENTS

(UFP-QAPP Manual Section 3.3.3)

The following sections outline the procedures that will be used to document project activities and sample collection, handling, tracking, and custody procedures during the investigation. All forms must be filled in as completely as possible.

Sample Nomenclature

Sample labeling will be conducted in general accordance with the procedures outlined in Resolution Consultant SOP-3-03A. Sample nomenclature put forth for this field event has been selected based on historical usage. The sample nomenclature includes the historical monitoring well identifier and the month and year the well was sampled (i.e., 01GW01GI36-MMY). Duplicate samples will be submitted to the laboratory as blind duplicates. The field QC blank codes that will be used during this LTM are as follows: EB for equipment rinsate blanks and TB for trip blanks. Field QC blanks will be labeled sequentially followed by the month, day, and year sampled (i.e., TBMMDDYY, EBMMDDYY, etc.). Samples to be used for matrix spike/matrix spike duplicate (MS/MSD) will be labeled MS/MSD on the container label and noted on the chain-of-custody; however, "MS/MSD" will not be part of the unique sample identifier in order to maintain consistency with the project database. Worksheet #18 provides anticipated sample identifiers for this scope of work.

Sample Collection Documentation

Documentation of field observations will be recorded in a field logbook and/or field log sheets including sample collection logs, boring logs, and monitoring well construction logs. Field logbooks utilized on this project will consist of a bound, water-resistant logbook. All pages of the logbook will be numbered sequentially and observations will be recorded with indelible ink.

Field sample log sheets will be used to document sample collection details and other observations and activities will be recorded in the field logbook. Instrument calibration logs will be used to record the daily instrument calibration.

For sampling and field activities, the following types of information will be recorded in the field logbook as appropriate:

- Site name and location
- Date and time of logbook entries
- Personnel and their affiliations
- Weather conditions



- Activities involved with the sampling
- Subcontractor activity summary
- Site observations including site entry and exit times
- Site sketches made onsite
- Visitor names, affiliations, arrival and departure times
- Health and safety issues, including PPE

Sample Handling and Tracking System

Following collection, all samples will be immediately placed on ice in a cooler. The glass sample containers will be enclosed in bubble-wrap to protect the bottle ware during shipment. The cooler will be secured using strapping tape along with a signed custody seal. Sample coolers will be delivered to a local courier location for priority overnight delivery to the selected laboratory for analysis. Samples will be preserved as appropriate based on the analytical method. The laboratories will provide pre-preserved sample containers for sample collection. Samples will be maintained at 0 to 6 degrees Celsius until delivery to the laboratory. Proper custody procedures will be followed throughout all phases of sample collection and handling.

After collection, each sample will be maintained in the sampler's custody until formally transferred to another party (e.g., FedEx). For all samples collected, chain-of-custody forms will document the date and time of sample collection, the sampler's name, and the names of all others who subsequently held custody of the sample. Specifications for chemical analyses will also be documented on the chain-of-custody form. Resolution Consultants SOP-3-03A provides further details on the chain-of-custody procedure.

These subsections outline the procedures that will be used by field and laboratory personnel to document project activities and sample collection procedures. All forms must be filled in as completely as possible.

Resolution Consultants personnel will collect the samples. The samplers will take care not to contaminate samples through improper handling. Samples will be sealed in appropriate containers, packaged by Resolution Consultants' personnel and placed into sealed coolers under chain-of-custody in accordance with the applicable SOP. All coolers will contain a temperature blank. Samples will be transferred under chain-of-custody to a courier as described below. Once received by the laboratory, receipt will be documented on the chain-of-custody form and the samples will be checked in. The samples will remain under chain-of-custody throughout the analysis period to ensure their integrity is preserved. Details are provided below.



Samples to be delivered to the laboratory(s) will be made by a public courier (i.e., FedEx). After samples have been collected, they will be sent to the laboratory(s) within 24 hours.

Field Sample Custody Procedures

Chain-of-custody protocols will be used throughout sample handling to establish the evidentiary integrity of sample containers. These protocols will be used to demonstrate that the samples were handled and transferred in a manner that would eliminate possible tampering. Samples for the laboratory will be packaged and shipped in accordance with Resolution Consultants SOP-3-04A.

A sample is under custody if:

- The sample is in the physical possession of an authorized person.
- The sample is in view of an authorized person after being in his/her possession.
- The sample is placed in a secure area by an authorized person after being in his/her possession.
- The sample is in a secure area, restricted to authorized personnel only.

Custody documentation is designed to provide documentation of preparation, handling, storage, and shipping of all samples collected. A multi-part form is used with each page of the form signed and dated by the recipient of a sample or portion of sample. The person releasing the sample and the person receiving the sample each will retain a copy of the form each time a sample transfer occurs.

Integrity of the samples collected will be the responsibility of identified persons from the time the samples are collected until the samples, or their derived data, are incorporated into the final report.

The Resolution Consultants FTL is responsible for the care and custody of the samples collected until they are delivered to the laboratory or are entrusted to a carrier. When transferring samples, the individuals relinquishing and receiving them will sign, date, and note the time on the chain-of-custody form. This record documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier). Upon arrival at the laboratory, internal sample custody procedures will be followed as defined in the Laboratory SOPs.



Laboratory Chain of Custody

Laboratory sample custody procedures (receipt of samples, archiving, and disposal) will be used according to Empirical SOPs. Coolers are received and checked for proper temperature. A sample cooler receipt form will be filled out to note conditions and any discrepancies. The chain-of-custody form will be checked against the sample containers for accuracy. Samples will be logged into the laboratory information management system and given a unique log number which can be tracked through processing. The laboratory project manager will notify the Resolution Consultants FTL verbally or via e-mail immediately if any problems are identified. Discrepancies and resolutions will be documented on the sample receiving checklist.



SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE

(UFP-QAPP Manual Section 3.4)

Matrix:		Groundwater				
Analytical Group:		Volatile Organic Compounds				
Analytical Method:		SW-846 8260B				
SOP Reference:		Empirical SOP 202				
QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory batch	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ. (see Box D-1 in QSM V4.2).	Correct problem; reanalyze any sample associated with a blank that fails criteria.	Analyst, Supervisor, QA Manager	Bias, Contamination	See Method/SOP QC Acceptance Limit Column
Surrogates	All field and QC samples	Surrogate DoD QSM Lab Limit¹	Reanalyze if sufficient sample is available. If reanalysis confirms failing recoveries, report and narrate.	Analyst, Supervisor, QA Manager	Accuracy, Bias	QC acceptance criteria specified in DoD QSM Version 4.2 See Method/SOP QC Acceptance Limit Column
		1,2-Dichloroethane-d4 70-120 70-120				
		4-Bromofluorobenzene 75-120 75-120				
		Dibromofluoromethane 85-115 85-115				
		Toluene-d8 85-120 85-120				
LCS	One LCS per preparatory batch	Analyte DoD QSM Lab Limit¹	Correct problem. If the LCS recoveries are high but the sample results are <LOQ narrate. Otherwise, re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Supervisor, QA Manager	Accuracy, Bias	QC acceptance criteria specified in DoD QSM Version 4.2 See Method/SOP QC Acceptance Limit Column
		Benzene 80-120 80-120				
		Chlorobenzene 80-120 80-120				
		Vinyl Chloride 50-145 50-145				



Matrix:		Groundwater				
Analytical Group:		Volatile Organic Compounds				
Analytical Method:		SW-846 8260B				
SOP Reference:		Empirical SOP 202				
QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Internal Standards	In all field samples and standards	Retention time \pm 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard or daily CCV.	Inspect MS or GC for malfunctions. Reanalyze all samples with internal standard failures. If reanalysis confirms matrix interference, report sample and narrate.	Analyst, Supervisor, QA Manager	Accuracy, Bias	Retention time \pm 30 seconds; EICP area within -50% to +100% of midpoint of ICAL
MS/MSD	One per preparatory batch per matrix	For matrix evaluation, use LCS recovery criteria; RPD \leq 30%.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy, Bias, Precision	For matrix evaluation, use LCS recovery criteria; RPD \leq 30%.

Notes:

¹ Laboratory limits are based on statistically-derived control charts provided by Empirical Laboratories, LLC (see Appendix E); however, DoD QSM limits will be used to qualify data.

VOCs	=	Volatile organic compounds	ICAL	=	Initial calibration
SOP	=	Standard operating procedure	EICP	=	Extracted ion current profile
QC	=	Quality control	MS	=	Mass Spectrometer
LOQ	=	Limit of quantitation	GC	=	Gas chromatograph
QA	=	Quality assurance	MS/MSD	=	Matrix spike/matrix spike duplicate
DoD QSM	=	Department of Defense Quality Systems Manual	RPD	=	Relative percent difference
LCS	=	Laboratory control sample			



SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (continued)

Matrix:		Groundwater and Surface water				
Analytical Group:		Metals				
Analytical Method:		SW-846 6010C				
SOP Reference:		Empirical SOP 105				
QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory batch	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).	Correct problem; re-prepare and/or reanalyze any sample associated with a blank that fails criteria.	Analyst, Supervisor, QA Manager	Bias, Contamination	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).
LCS	One LCS per preparatory batch	Analyte	Correct problem. If the LCS recoveries are high but the sample results are <LOQ narrate. Otherwise, re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Supervisor, QA Manager	Accuracy, Bias	QC acceptance criteria specified in DoD QSM Version 4.2 See Method/SOP QC Acceptance Limit Column
		DoD QSM				
		Lab Limit¹				
Sample Duplicate or MSD	One per preparatory batch	RPD ≤ 20%	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Precision	RPD ≤ 20%
Matrix Spike	One per preparatory batch per matrix	For matrix evaluation, use LCS recovery criteria	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy, Bias	For matrix evaluation, use LCS recovery criteria



Matrix:		Groundwater and Surface water				
Analytical Group:		Metals				
Analytical Method:		SW-846 6010C				
SOP Reference:		Empirical SOP 105				
QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Dilution Test	One per preparatory batch	Five-fold dilution must agree within $\pm 10\%$ of the original measurement for samples with concentrations $> 50 \times \text{LOQ}$	Perform Post Digestion Spike	Analyst, Supervisor, QA Manager	Accuracy, Bias	Five-fold dilution must agree within $\pm 10\%$ of the original measurement for samples with concentrations > 50 times LOQ
Post Digestion Spike	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if possible).	Recovery 80-120%	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy, Bias	Recovery 80-120%

Notes:

¹ Laboratory limits are based on statistically-derived control charts provided by Empirical Laboratories, LLC (see Appendix E); however, DoD QSM limits will be used to qualify data

SOP = Standard operating procedure
 QC = Quality control
 LOQ = Limit of quantitation
 QA = Quality assurance
 LCS = Laboratory control sample

DoD QSM = Department of Defense Quality Systems Manual
 ICAL = Initial calibration
 MSD = Matrix spike duplicate
 RPD = Relative percent difference
 MSD = Matrix spike duplicate



SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (continued)

Matrix:		Groundwater				
Analytical Group:		Alkalinity				
Analytical Method:		SM 2320B				
SOP Reference:		Empirical SOP 154				
QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples.	Analyte concentration must be < LOQ.	Correct problem, re-prepare and reanalyze along with all associated samples.	Analyst, Supervisor, QA Manager	Bias/Contamination	No analytes detected > LOQ
LCS	One per preparatory batch of 20 or fewer samples.	%R must be within 80-120% of true value.	Correct problem. If the LCS recoveries are high but the sample results are <LOQ narrate. Otherwise, re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Supervisor, QA Manager	Accuracy/Bias	%R must be within 80-120% of true value.
Laboratory Duplicate	One per batch of 20 or fewer samples.	RPD \leq 20%	Correct problem and reanalyze sample and duplicate.	Analyst, Supervisor, QA Manager	Precision	RPD \leq 20 %

Notes:

SOP	=	Standard operating procedure	LCS	=	Laboratory control sample
QC	=	Quality control	%R	=	Percent recovery
LOQ	=	Limit of quantitation	MS/MSD	=	Matrix spike/matrix spike duplicate
QA	=	Quality assurance	RPD	=	Relative percent difference



SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (continued)

Matrix:		Groundwater				
Analytical Group:		Chloride, Sulfate				
Analytical Method:		E300.0				
SOP Reference:		Empirical SOP 145				
QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per batch of up to 20 samples.	Analyte concentration must be $< \frac{1}{2}$ LOQ.	Correct problem, re-prepare, and reanalyze along with all associated samples.	Analyst, Supervisor, QA Manager	Bias/Contamination	No analytes detected $> \frac{1}{2}$ LOQ and $> \frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. (see Box D-1 in QSM V4.2).
LCS	One per batch of up to 20 samples.	%R must be within 80-120% of true value.	Correct problem. If the LCS recoveries are high but the sample results are $< \text{LOQ}$ narrate. Otherwise, re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Supervisor, QA Manager	Accuracy/Bias	%R must be within 80-120% of true value.
Laboratory Duplicate	One per every 20 samples.	RPD must be $\leq 10\%$.	Correct problem, reanalyze sample, and duplicate.	Analyst, Supervisor, QA Manager	Precision	RPD must be $\leq 20\%$.
MS/MSD	One per batch of up to 20 samples.	%R should be within 80-120% of true value. RPD should be $\leq 10\%$.	Corrective action will not be taken for samples when recoveries are outside limits and LCS criteria are met. If both the LCS and MS/MSD %Rs are unacceptable, then re-prepare and reanalyze the samples and QC.	Analyst, Supervisor, QA Manager	Accuracy/Bias Precision	%R should be within 80-120% of true value. RPD should be $\leq 20\%$.

Notes:

SOP = Standard operating procedure
 QC = Quality control
 LOQ = Limit of quantitation
 QA = Quality assurance
 DL = Detection limit

LCS = Laboratory control sample
 %R = Percent recovery
 MS/MSD = Matrix spike/matrix spike duplicate
 RPD = Relative percent difference



SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (continued)

Matrix:		Groundwater				
Analytical Group:		Methane, Ethane, Ethene				
Analytical Method:		RSK-175				
SOP Reference:		Empirical SOP 236				
QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per batch of 20 or less.	Result must be < LOQ.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results	Analyst, Supervisor, QA Manager	Bias/Contamination	Result must be < LOQ.
LCS	One per batch of 20 or less.	%R must be within 70-130%.	Correct problem. If the LCS recoveries are high but the sample results are <LOQ narrate. Otherwise, re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Lab internal limits for %R. %R must be within 70-130%.
MS/MSD	One per batch of 20 or less.	%R should be within 70-130%, RPD should be ≤ 30%	Check for errors in calculations and spike preparation. Check un-spiked sample results and surrogate recoveries for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated.	Analyst, Supervisor, QA Manager	Precision/Accuracy/Bias	Lab internal limits for %R and RPD. %R should be within 70-130%, RPD should be ≤ 30%

Notes:

SOP = Standard operating procedure
 QC = Quality assurance
 LOQ = Limit of quantitation
 QA = Quality assurance

LCS = Laboratory control sample
 %R = Percent recovery
 MS/MSD = Matrix spike/matrix spike duplicate
 RPD = Relative percent difference



SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (continued)

Matrix:		Groundwater				
Analytical Group:		Total Organic Carbon				
Analytical Method:		SW-846 9060A				
SOP Reference:		Empirical SOP 221				
QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples.	Analyte concentration must be < ½ LOQ.	Correct problem, re-prepare and reanalyze along with all associated samples.	Analyst, Supervisor, QA Manager	Bias/Contamination	No analytes detected > ½ LOQ
LCS	One per preparatory batch of 20 or fewer samples.	%R must be within 80-120% of true value.	Correct problem. If the LCS recoveries are high but the sample results are < LOQ narrate. Otherwise, re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Supervisor, QA Manager	Accuracy/Bias	%R must be within 80-120% of true value.
MS/MSD	One per preparatory batch of 20 or fewer samples per matrix.	%R should be within 75-125% of true value. RPD should be ≤20%.	Corrective action will not be taken for samples when recoveries are outside limits and LCS criteria are met. If both the LCS and MS/MSD %Rs are unacceptable, then re-prepare and reanalyze the samples and QC.	Analyst, Supervisor, QA Manager	Accuracy/Bias/Precision	%R should be within 80-120% of true value. RPD should be ≤20%.
Laboratory Duplicate	One per batch of 20 or fewer samples, sample dup is not required if MSD is performed.	RPD ≤20% for concentrations > 5 times the LOQ	Correct problem and reanalyze sample and duplicate.	Analyst, Supervisor, QA Manager	Precision	RPD ≤20% for concentrations > 5 times the LOQ

Notes:

SOP = Standard operating procedure
 QC = Quality control
 LOQ = Limit of quantitation
 QA = Quality assurance

LCS = Laboratory control sample
 %R = Percent recovery
 MS/MSD = Matrix spike/matrix spike duplicate
 RPD = Relative percent difference



SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (continued)

Matrix:		Groundwater				
Analytical Group:		Dissolved Gases (Hydrogen and Carbon Disulfide)				
Analytical Method:		Pace (Microseeps) AM20GAX				
SOP Reference:		Pace (Microseeps) AM20GAX				
QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples.	Analyte concentration must be $< \frac{1}{2}$ LOQ.	Determine the source of the error, notify the client, and narrate appropriately.	Analyst, Laboratory Department Manager, and Data Validator	Bias/Contamination	Analyte concentration must be $< \frac{1}{2}$ LOQ.
Calibration Blank	At the beginning of analytical sequence, after every 15 samples, and at the end of the sequence.	Analyte concentration must be $< \frac{1}{2}$ LOQ.	Correct problem, re-prepare, and reanalyze along with all associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Bias/Contamination	Analyte concentration must be $< \frac{1}{2}$ LOQ.
LCS	One per preparatory batch of 20 or fewer samples.	%R must be within 80-120% of true value.	Re-prepare, and reanalyze the LCS along with all associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	%R must be within 80-120% of true value.

Notes:

QC	=	Quality control
SOP	=	Standard operating procedure
LOQ	=	Limit of quantitation
DL	=	Detection limit
LCS	=	Laboratory control sample
%R	=	Percent recovery
RPD	=	Relative percent difference



SAP WORKSHEET #29: PROJECT DOCUMENTS AND RECORDS TABLE

(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
<i>Sample Collection Documents and Records</i> Project personnel sign-off record Field logbook (and sampling notes) Field sample forms (e.g., sample log sheets, drilling logs, etc.) Chain-of-custody records Sample shipment air bills Equipment calibration logs Photographs Sampling and Analysis Plans (SAP) including field sampling standard operating procedures (SOPs) Safe work permit forms	Sample collection documents and records (may include printed copy as well as electronic information) will be maintained at the Resolution Consultants office at 5724 Summer Trees Drive, Memphis, Tennessee 38134. These files will be retained for fifty (50) years after the last decision document is signed for NAS Pensacola.
<i>Analytical Results Documents and Records</i> Sample receipt/log-in forms Sample preparation logs Equipment calibration logs Sample analysis run logs Reported field sample results Reported results for standards, quality control checks Reported results for standards, quality control samples Data completeness checklists Data validation memoranda	<p>Analytical results, documents and records will be provided by the laboratory in printed and electronic formats. Although available in the Administrative Record file, laboratory reports are typically filed at a separate location and are available upon request. Printed copies of laboratory data will be stored at a third-party secure professional document storage firm until transfer to the Federal Records Center (FRC). The records will be transferred to FRC 3 years after completion of a response action. These files will be retained for fifty (50) years after the last decision document is signed for NAS Pensacola.</p> <p>Electronic analytical results will also be verified, entered, and maintained in a database on a password protected Structured Query Language server. Data qualifiers will be added to the database during data validation. After validation, the validated data files will be transferred to the Navy's Naval Installation Restoration Information Solution (NIRIS) data management system.</p>



Document	Where Maintained
<p><i>Other Documents</i> Personnel training records Health and Safety certifications Health and Safety Plan Field Sampling Audit Checklist Letter reports, Annual OU1 Long Term Monitoring Reports, etc. Analytical Audit Checklist</p>	<p>Personnel training records and health and safety certificates will be stored in personnel records and electronically in the Resolution Consultant's training database located at project file at 5724 Summer Trees Drive, Memphis, Tennessee 38134.</p> <p>Plans and reports will be stored in printed version and electronically in the Administrative Record file. Printed copies will be stored at a third-party secure professional document storage firm until transfer to the FRC. The records will be transferred to FRC 3 years after completion of a response action. These files will be retained for fifty (50) years after the last decision document is signed for NAS Pensacola.</p> <p>Field Audit Checklists are not considered part of the Administrative Record file and will be stored in the Resolution Consultants project file at 5724 Summer Trees Drive, Memphis, Tennessee 38134, and electronically in the server library.</p> <p>Analytical Audit Checklists will be retained by the respective accreditation authorities.</p>
<p><i>Final Document/Records Repository</i> Administrative Record files Site files Post decision Files Analytical data Spatial data Maps</p>	<p>All final documents/Records repositories will be stored in accordance with in the NAVFAC Environmental Restoration Recordkeeping Manual. Printed copies will be stored at a third-party secure professional document storage firm until transfer to the FRC, and electronic copies will be maintained, verified, and stored on the Navy's NIRIS data management system. The records will be transferred to FRC 3 years after completion of a response action. These files will be retained for fifty (50) years after the last decision document is signed for NAS Pensacola.</p>



SAP WORKSHEET #30: ANALYTICAL SERVICES TABLE

(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ID Numbers	Empirical Laboratory Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (name and address, contact person and telephone number)	Backup Laboratory/Organization
Groundwater	Select Volatile Organic Compounds	See Worksheet #18	Empirical SOP-202	21 Days	Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270, Nashville, Tennessee 37228. Sonya Gordon, sgordon@empirlabs.com, 615-345-1115	None
Groundwater	Select Metals	See Worksheet #18	Empirical SOP-100/105	21 Days	Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270, Nashville, Tennessee 37228. Sonya Gordon, sgordon@empirlabs.com, 615-345-1115	None
Surface Water	Total iron	See Worksheet #18	Empirical SOP-100/105	21 Days	Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270, Nashville, Tennessee 37228. Sonya Gordon, sgordon@empirlabs.com, 615-345-1115	None
Groundwater	Geochemical — alkalinity	See Worksheet #18	Empirical SOP-154	21 Days	Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270, Nashville, Tennessee 37228. Sonya Gordon, sgordon@empirlabs.com, 615-345-1115	None
Groundwater	Geochemical — chloride, nitrate, sulfate	See Worksheet #18	Empirical SOP-145	21 Days	Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270, Nashville, Tennessee 37228. Sonya Gordon, sgordon@empirlabs.com, 615-345-1115	None
Groundwater	Geochemical — methane, ethane, ethene	See Worksheet #18	Empirical SOP-236	21 Days	Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270, Nashville, Tennessee 37228. Sonya Gordon, sgordon@empirlabs.com, 615-345-1115	None
Groundwater	Geochemical — total organic carbon	See Worksheet #18	Empirical SOP-221	21 Days	Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270, Nashville, Tennessee 37228. Sonya Gordon, sgordon@empirlabs.com, 615-345-1115	None
Groundwater	Hydrogen and Carbon Dioxide	See Worksheet #18	Pace (Microseeps) SOP AM20GAX	21-Days	Pace Analytical Energy Services, LLC (Microseeps, Inc.), 220 William Pitt Way, Pittsburgh, Pennsylvania 15238. Robbin Robl, rrobl@microseeps.com, 412-826-5245	None

Notes:

SOP = standard operating procedure
 ID = identification



SAP WORKSHEET #31: PLANNED PROJECT ASSESSMENTS TABLE

(UFP-QAPP Manual Section 4.1.1)

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment	Person(s) Responsible for Responding to Assessment Findings	Person(s) Responsible for Identifying and Implementing Corrective Action	Person(s) Responsible for Monitoring Effectiveness of Corrective Action
Onsite Laboratory Systems Audit	Every 18 months	External	DoD ELAP	DoD ELAP Auditor	Marcia McGinnity, QAO, Empirical Laboratories	Marcia McGinnity, QAO, Empirical Laboratories	Marcia McGinnity, QAO, Empirical Laboratories
Onsite Laboratory Systems Audit	Every 2 years	External	NELAP	NELAP Auditor	Marcia McGinnity, QAO, Empirical Laboratories Patrick McLoughlin Pace (Microseeps, Inc.)	Marcia McGinnity, QAO, Empirical Laboratories Patrick McLoughlin Pace (Microseeps, Inc.)	Marcia McGinnity, QAO, Empirical Laboratories Patrick McLoughlin Pace (Microseeps, Inc.)
Onsite Field Sampling Systems Audit	One per contract year	Internal	Resolution Consultants	Tina Cantwell, QAO (or designee), Resolution Consultants	Allison Harris, TOM Resolution Consultants	Tina Cantwell, QAO and Allison Harris, TOM Resolution Consultants	Tina Cantwell, QAO and Allison Harris, TOM Resolution Consultants
Onsite Health and Safety Audit	One per contract year	Internal	Resolution Consultants	John Knopf, HSM (or designee), Resolution Consultants	Allison Harris, TOM Resolution Consultants	John Knopf, HSM and Allison Harris, TOM Resolution Consultants	John Knopf, HSM and Allison Harris, TOM Resolution Consultants

Notes:

DoD ELAP = Department of Defense Environmental Laboratory Accreditation Program
QAO = Quality assurance officer
NELAP = National Environmental Laboratory Accreditation Program
TOM = Task order manager
HSM = Health and safety manager



SAP WORKSHEET #32: ASSESSMENT FINDINGS AND CORRECTIVE ACTION RESPONSES TABLE

(UFP-QAPP Manual Section 4.1.2)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response	Timeframe for Response
Laboratory Systems Audit	Verbal debriefing, Written audit report	Marcia McGinnity, QAO, Empirical Laboratories Patrick McLoughlin Pace (Microseeps, Inc.)	Not specified by DoD ELAP or NELAP	Corrective action plan, Letter	DoD ELAP and NELAP Accrediting Bodies	Specified by DoD ELAP and NELAP Accrediting Bodies
Field Sampling Systems Audit	Audit checklist and written audit finding summary	Allison Harris, TOM, Resolution Consultants; William Honea, FTL, Resolution Consultants	Dependent on findings; if major, a stop work may be issued immediately; however, if minor, within 1 week of audit	Written memo	Tina Cantwell, QAO, Resolution Consultants	Within 21 days of notification
Health and Safety Audit	Audit checklist and written audit finding summary	Allison Harris, TOM, Resolution Consultants	Dependent on findings; if major, a stop work may be issued immediately; however, if minor, within 1 week of audit	Written memo	John Knopf, HSM, Resolution Consultants	Within 21 days of notification
Field Supervision	Site log book and sample collection logs	Allison Harris, TOM, Resolution Consultants; William Honea, FTL, Resolution Consultants	Immediately, when discovered	Entry in site log book, potential retraining	Allison Harris, TOM, Resolution Consultants; William Honea, FTL, Resolution Consultants	Within 24 hours

Notes:

Assessment findings will be communicated with appropriate staff during verbal audit debriefings and in the written audit report and will be documented on audit checklists, audit reports, and the Annual OU1 Long Term Monitoring Report submitted to the regulatory agencies.

DoD ELAP = Department of Defense Environmental Laboratory Accreditation Program

QAO = Quality assurance officer

NELAP = National Environmental Laboratory Accreditation Program

TOM = Task order manager

FTL = Field team leader

HSM = Health and safety manager



SAP WORKSHEET #33: QUALITY ASSURANCE MANAGEMENT REPORTS TABLE

(UFP QAPP Manual Section 4.2)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data Validation	Report per data package	Within 4 weeks of receipt of laboratory data	Resolution Consultants project chemist or designee	TOM, project file, Resolution Consultants
Major Analysis Problem Identification (Internal Resolution Consultants Memorandum)	When persistent analysis problems are detected by Resolution Consultants that may impact data usability	Immediately upon detection of problem (same day)	Resolution Consultants quality assurance officer or project chemist	TOM, program manager, contracts department, project file, Resolution Consultants
Progress Report	Monthly for duration of the project	Monthly	TOM, Resolution Consultants	Navy RPM, program manager, project file, Resolution Consultants
Laboratory Quality Assurance Report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem (same day)	Laboratory quality assurance manager or project manager, Empirical Laboratory	TOM, project chemist, project file, Resolution Consultants

Notes:

TOM = Task order manager
 RPM = Remedial project manager



SAP WORKSHEETS #34-36: DATA VERIFICATION AND VALIDATION (STEPS I AND IIA/IIB) PROCESS TABLE

(UFP-QAPP Manual Section 5.2.1), (UFP-QAPP Manual Section 5.2.2), (Figure 37 UFP-QAPP Manual), (Table 9 UFP-QAPP Manual)

Data Review Input	Description	Responsible for Verification (name, organization)	Step I/IIa/ IIB ¹	Internal/ External ²
Verification Chain-of-custody forms Sample Login/ Receipt	Review the sample shipment for completeness, integrity, and sign accepting the shipment. All sample labels will be checked against the chain-of-custody form, and any discrepancies will be identified, investigated, and corrected. The samples will be logged in at every storage area and work station required by the designated analyses. Individual analysts will verify the completeness and accuracy of the data recorded on the forms.	Laboratory sample custodians and analysts, Empirical Laboratory	I	Internal
Verification Chain-of-custody forms	Check that the chain-of-custody form was signed/dated by the sampler relinquishing the samples and by the laboratory sample custodian receiving the samples for analyses.	Project chemist or data validators, Resolution Consultants	I	External
Verification SAP sample tables	Verify that all proposed samples listed in the SAP tables have been collected.	FTL or designee, Resolution Consultants	I	External
Verification Sample log sheets and field notes	Verify that information recorded in the log sheets and field notes are accurate and complete.	FTL or designee, Resolution Consultants	I	External
Verification Field QC samples	Check that field QC samples, described in Worksheet #12 and listed in Worksheet #20 were collected as required.	FTL or designee, Resolution Consultants	I	External
Verification Analytical data package	Verify all analytical data packages will be verified internally for completeness by the laboratory performing the work. The laboratory project manager (or designee) will sign the case narrative for each data package.	Laboratory project manager, Empirical Laboratory	I	Internal
Verification Analytical data package	Verify the data package for completeness. Missing information will be requested from the laboratory and validation (if performed) will be suspended until missing data are received.	FTL, project chemist or data validators, Resolution Consultants	I	External
Verification Electronic data deliverables	Verify the electronic data against the chain-of-custody and hard copy data package for accuracy and completeness.	Data manager and/or validator, Resolution Consultants	I	External
Validation Chain-of-custody	Examine the traceability of the data from time of sample collection until reporting of data. Ensure that the custody and integrity of the samples were maintained from collection to analysis and the custody records are complete and any deviations are recorded.	Project chemist or data validators, Resolution Consultants	IIa	External
Validation Holding Times	Review that the samples were shipped and stored at the required temperature and sample pH for chemically-preserved samples meet the requirements listed in Worksheet #19. Ensure that the analyses were performed within the holding times. If holding times were not met, confirm that deviations were documented.	Project chemist or data validators, Resolution Consultants	IIa	External



Data Review Input	Description	Responsible for Verification (name, organization)	Step I/IIa/ IIb ¹	Internal/ External ²
Validation Laboratory data results for accuracy	Ensure that the laboratory QC samples were analyzed and that the MPC, listed in Worksheet #28, were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed, as listed in Worksheet #12, and that the analytical QC criteria were met.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
Validation Field and laboratory duplicate analyses for precision	Check the field sampling precision by calculating the RPD for field duplicate samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and LCS/LCSDs. Ensure compliance with the precision goals listed in Worksheet #12 and 28.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
Validation Sample results for representativeness	Check that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
Validation Project action limits	Assess and document the impact on matrix interferences or sample dilutions performed because of the high concentration of one or more contaminant, on the other target compounds reported as undetected.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
Validation Data quality assessment report	Summarize deviations from methods, procedures, or contracts. Qualify data results based on method or QC deviation and explain all the data qualifications. Present tabular qualified data and data qualifier codes and summarize data qualification outliers. Determine if the data met the MPC and determine the impact of any deviations on the technical usability of the data.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
Validation SAP QC sample documentation	Ensure that all QC samples specified in the SAP were collected and analyzed and that the associated results were within acceptance limits.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
Validation Analytical data deviations	Determine the impact of any deviation from sampling or analytical methods and laboratory SOP requirements and matrix interferences effect on the analytical results.	Project chemist or data validators, Resolution Consultants	IIb	External
Validation Project quantitation limits for sensitivity	Ensure that the project detection limits were achieved.	Project chemist or data validators, Resolution Consultants	IIb	External
Validation Groundwater — Select VOCs: benzene, chlorobenzene, and vinyl chloride	<p>Assess VOC data against MPC identified in Worksheets #12, 19, 24, and 28. <i>U.S. EPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review</i>, (June 2008) will be used as a guidance on applying qualifiers when MPC identified in Worksheets #12, 19, 24, and 28 are not met, including identifying when samples will be qualified estimated or rejected and when individual or all samples in a batch will be qualified.</p> <p>As described in Worksheet #14, checklists will not be used for validation because it will be performed using a combination of electronic automated data review and data package review. All findings will be documented on the DVA workbook and in the data validation report.</p>	Project chemist or data validators, Resolution Consultants	IIa/IIb	External



Data Review Input	Description	Responsible for Verification (name, organization)	Step I/IIa/ IIb ¹	Internal/ External ²
Validation Groundwater, Surface Water — Select metals: nickel and iron	<p>Assess metals data against MPC identified in Worksheets #12, 19, 24, and 28. <i>U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review</i>, (January 2010) will be used as a guidance on applying qualifiers when MPC identified in Worksheets #12, 19, 24, and 28 are not met, including identifying when samples will be qualified estimated or rejected and when individual or all samples in a batch will be qualified.</p> <p>As described in Worksheet #14, checklists will not be used for validation because it will be performed using a combination of electronic automated data review and data package review. All findings will be documented on the DVA workbook and in the data validation report.</p>	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
Validation Groundwater — Organic Geochemical parameters: methane, ethane, ethene, hydrogen, and carbon dioxide	<p>Assess organic geochemical data against MPC identified in Worksheets #12, 19, 24, and 28. <i>U.S. EPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review</i>, (June 2008) will be used as a guidance on applying qualifiers when MPC identified in Worksheets #12, 19, 24, and 28 are not met, including identifying when samples will be qualified estimated or rejected and when individual or all samples in a batch will be qualified. The end use of the geochemical data will be used as a screening level tool to assess remedial effectiveness; therefore, validation for geochemical data will be limited to the following elements: holding times, blank analyses, and laboratory control samples.</p> <p>As described in Worksheet #14, checklists will not be used for validation because it will be performed using a combination of electronic automated data review and data package review. All findings will be documented on the DVA workbook and in the data validation report.</p>	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
Validation Groundwater — Inorganic Geochemical Parameters: nitrate, sulfate, chloride, total organic carbon, and alkalinity	<p>Assess inorganic geochemical data against MPC identified in Worksheets #12, 19, 24, and 28. <i>U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review</i>, (January 2010) will be used as a guidance on applying qualifiers when MPC identified in Worksheets #12, 19, 24, and 28 are not met, including identifying when samples will be qualified estimated or rejected and when individual or all samples in a batch will be qualified. The end use of the geochemical data will be used as a screening level tool to assess remedial effectiveness; therefore, validation for geochemical data will be limited to the following elements: holding times, blank analyses, and laboratory control samples.</p> <p>As described in Worksheet #14, checklists will not be used for validation because it will be performed using a combination of electronic automated data review and data package review. All findings will be documented on the DVA workbook and in the data validation report.</p>	Project chemist or data validators, Resolution Consultants	IIa/IIb	External



Data Review Input	Description	Responsible for Verification (name, organization)	Step I/IIa/ IIb ¹	Internal/ External ²																																													
Validation Data qualifiers	Qualifiers that will be applied during the data validation process are summarized below and, as indicated, results will be considered usable for interpretation. Use of rejected data will be decided by the project team.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External																																													
	<table><tr><th>Data Qualifier</th><th>Qualifier Definition</th><th>Interpret Result As a Detection?</th><th>Result Usable?</th><th>Potential Result Bias</th></tr><tr><td>no qualifier</td><td>Acceptable</td><td>Yes</td><td>Yes</td><td>None expected</td></tr><tr><td>I</td><td>Estimated</td><td>Yes</td><td>Yes</td><td>High or Low</td></tr><tr><td>IJ</td><td>Estimated</td><td>Yes</td><td>Yes</td><td>High or Low</td></tr><tr><td>J</td><td>Estimated</td><td>Yes</td><td>Yes</td><td>High or Low</td></tr><tr><td>U</td><td>Undetected</td><td>No</td><td>Yes</td><td>None expected</td></tr><tr><td>UJ</td><td>Undetected and Estimated</td><td>No</td><td>Yes</td><td>High or Low</td></tr><tr><td>UR</td><td>Undetected and Rejected</td><td>No</td><td>No</td><td>Unspecified</td></tr><tr><td>R</td><td>Rejected</td><td>No</td><td>No</td><td>Unspecified</td></tr></table>				Data Qualifier	Qualifier Definition	Interpret Result As a Detection?	Result Usable?	Potential Result Bias	no qualifier	Acceptable	Yes	Yes	None expected	I	Estimated	Yes	Yes	High or Low	IJ	Estimated	Yes	Yes	High or Low	J	Estimated	Yes	Yes	High or Low	U	Undetected	No	Yes	None expected	UJ	Undetected and Estimated	No	Yes	High or Low	UR	Undetected and Rejected	No	No	Unspecified	R	Rejected	No	No	Unspecified
	Data Qualifier				Qualifier Definition	Interpret Result As a Detection?	Result Usable?	Potential Result Bias																																									
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	U				Undetected	No	Yes	None expected																																									
	UJ				Undetected and Estimated	No	Yes	High or Low																																									
	UR				Undetected and Rejected	No	No	Unspecified																																									
R	Rejected	No	No	Unspecified																																													
Note: Both the I and J qualifiers indicate an estimated value. The I-qualifier, applied by the laboratory, is defined by Florida Department of Environmental Protection as the result is an estimated value between the detection limit and the quantitation limit. The I-qualifier, applied by the laboratory, will remain on the result to provide the end user additional information that the value is estimated below the quantitation limit. The J-qualifier will be applied during validation to indicate a value is estimated.																																																	

Notes:

¹ IIa = Compliance with methods, procedures, and contracts [see Table 10, page 117, UFP-QAPP manual, V.1, March 2005.]

¹ IIb = Comparison with measurement performance criteria in the SAP [see Table 11, page 118, UFP-QAPP manual, V.1, March 2005]

² Internal or external in relation to the data generator.

SAP = Sampling and analysis plan

FTL = Field team leader

QC = Quality control

RPD = Relative percent difference

MS/MSD = Matrix spike/Matrix Spike duplicate

LCS/LCSD = Laboratory control sample/laboratory control sample duplicate

SOP = Standard operating procedure

VOC = Volatile organic compounds

U.S. EPA = U.S. Environmental Protection Agency

MPC = Measurement performance criteria

DVA = Data Validation Assistant

SAP WORKSHEET #37: USABILITY ASSESSMENT

(UFP-QAPP Manual Section 5.2.3)

Data Review

The usability of the data directly affects whether project objectives can be achieved. Data usability will be assessed using the guidance provided in *Process for Assessing Data Usability* (FDEP 2008) and the characteristics described below will be evaluated at a minimum. The results of these evaluations will be included in the project report. To the extent required by the type of data being reviewed, the assessors will consult with other technically competent individuals to render sound technical assessments of these data characteristics:

- **Completeness** — Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount expected to be obtained under correct normal conditions. It is expected that 100% of the planned sampling points will be collected. The completeness goal for field measurements will be greater than 90%. Laboratory analysis for this project will have a completeness goal greater than 95% to account for unanticipated results that may be rejected during data validation. Completeness can be calculated using the following equation.

$$\%Completeness = \frac{\text{No. of Valid Tests}}{\text{Total Tests Taken}} \times 100$$

The FTL, acting on behalf of the Project Team, will determine whether deviations from the scheduled sample collection or analyses occurred. If they have occurred and the Resolution Consultants TOM determines that the deviations compromise the ability to meet project objectives she will consult with the Navy RPM and other project team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions.

- **Precision** — Precision measures the reproducibility of measurements and methods, and is defined for qualitative data as a group of values' variability compared with its average value. To assess the precision of the measurement systems used in this project, field duplicates will be obtained and analyzed with the samples collected. Precision of laboratory analysis will be assessed by comparing the relative percent difference (RPD) of analytical results between MS and MSDs (or sample duplicates) and the measurement quality objectives will be those cited in Worksheets #12 and #28. The RPD will be calculated for each pair of duplicate analysis using the following equation:

$$RPD = \frac{(S - D)}{(S + D) / 2} \times 100$$

Where:

S = sample result
D = duplicate result

The project chemist, acting on behalf of the Project Team, will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in Worksheets #12 and #28. This will also include a comparison of field and laboratory precision with the expectation that laboratory duplicate results will be no less precise than field duplicate results. If the goals are not met or data have been flagged as estimated (J qualifier) limitations on the use of the data will be described in the project report.

- **Accuracy** — Accuracy is the degree to which a given result agrees with the true value. The accuracy of an entire measurement system is an indication of any bias that exists. Spiked sample results provide information needed to assess the accuracy of analyses. Specifically, surrogate spike, MS/MSD, and laboratory control sample (LCS) percent recoveries (%Rs) are used to assess accuracy. Every organic sample is spiked with known quantities of non-target surrogate compounds. Five percent of all samples analyzed are spiked with target chemicals for the MS/MSD (or sample duplicates). If the calculated %Rs for the known spike concentrations are within defined control limits set by each method, the reported sample concentrations are considered accurate. The accuracy measurement quality objectives will be those cited in Worksheets #12 and #28. Accuracy is calculated using the following equation:

$$\% R = \frac{(SSR - SR)}{SA} \times 100$$

Where:

SSR = spike sample recovery
SR = sample recovery
SA = concentration of spike added

The project chemist, acting on behalf of the Project Team, will determine whether the accuracy/bias goals were met for project data. This assessment will include an evaluation of field and laboratory contamination; instrument calibration variability; and analyte recoveries for surrogates, matrix spike, matrix spike duplicate, and laboratory control samples against the



goals identified in worksheets #24 and #28. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

- Representativeness — A project scientist, identified by the Resolution Consultants TOM and acting on behalf of the Project Team, will determine whether the data are adequately representative of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and analyzed in accordance with this SAP, by reviewing spatial and temporal data variations, and by comparing these characteristics to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the project scientist indicates that a quantitative analysis is required.
- Comparability — The project chemist, acting on behalf of the Project Team, will determine whether the data generated under this project are sufficiently comparable to historical property data generated by different methods and for samples collected using different procedures and under different property conditions. This will be accomplished by comparing overall precision and bias among data sets for each matrix and analytical fraction. This will not require quantitative comparisons unless the project chemist indicates that such quantitative analysis is required.
- Sensitivity — The project chemist, acting on behalf of the Project Team, will determine whether project sensitivity goals listed in Worksheet #15 are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described.

Describe the evaluative procedures used to assess overall measurement error associated with the project:

After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. In addition to the evaluations described above, a series of inspections and statistical analyses will be performed to estimate these characteristics. The statistical evaluations will include simple summary statistics for target analytes, such as maximum concentration, minimum concentration, number of samples

exhibiting non-detected results, number of samples exhibiting positive results, and the proportion of samples with detected and non-detected results. The Project Team members, identified by the Resolution Consultants TOM, will assess whether the data collectively support the attainment of project objectives. They will consider whether any missing or rejected data have compromised the ability to make decisions or to make the decisions with the desired level of confidence. The data will be evaluated to determine whether missing or rejected data can be compensated by other data or whether additional data collection is necessary.

Identify the personnel responsible for performing the usability assessment:

The Resolution Consultants TOM, project chemist, and FTL will be responsible for conducting the listed data usability assessments. The data usability assessment will include the elements provided in Table 37-1.

Table 37-1 Data Usability Process	
Step 1	Review the project's objectives and sampling design The sampling design, data quality objectives, and measurement performance criteria provided in this SAP will be reviewed to assess that they are still applicable.
Step 2	Review the data verification and validation outputs which will include: <ul style="list-style-type: none"> Review available quality assurance reports, including the data verification and data validation reports. Summarize data into tables and trend charts and assess patterns and anomalies. Review deviations from planned activities (e.g., number and locations of samples, holding time exceedances, damaged samples, and procedural deviations) and assess their impacts on the data usability. Evaluate implications of unacceptable quality control sample results.
Step 3	Verify the assumptions of the selected statistical method which will include: Verifying whether underlying assumptions for the Mann Kendall statistical method are valid. Common assumptions include the distributional form of the data, independence of the data, dispersion characteristics, homogeneity, etc. Minor deviations from assumptions will not be considered critical to statistical analysis and data interpretation. However, if serious deviations from assumptions are discovered, then another statistical method may need to be selected.
Step 4	Implement the statistical method Data analysis will include: <ul style="list-style-type: none"> Performing Mann Kendall statistical analysis Plotting on trend charts (concentration versus time) Data summary with comparison against screening levels identified on worksheet #15 Using these data analysis tools, data will be evaluated to assess whether the underlying assumptions hold, or whether departures are acceptable, given the actual data and other information.
Step 5	Document data usability and draw conclusions Data usability will be documented in the annual Long-Term Monitoring Reports and will include: <ul style="list-style-type: none"> Assessment on whether data can be used as intended, considering implications of deviations and corrective actions. Discussion of data quality indicators and identification of data use limitations. Sampling design performance evaluation. Conceptual site model update (if warranted) and document conclusions.



The data usability assessment will be reviewed with the Project Team. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face to face meeting or a teleconference depending on the extent of identified deficiencies. If no significant deficiencies are identified, the data usability assessment will simply be documented in the project report and reviewed during the normal document review cycle.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

The data will be presented in tabular format, including data qualifications such as estimation (J, UJ) or rejection (R). The project report will identify and describe the data usability limitations and suggest re-sampling or other corrective actions, if necessary. Graphical presentations of the data such as concentration tag maps will be generated as part of the overall data evaluation process.



REFERENCES

- Battelle. *User Guide Department of the Navy Guidance for Planning and Optimizing Monitoring Strategies*. Battelle; Columbus, Ohio. (2008).
- Bechtel Environmental, Inc. *Long-Term Groundwater Monitoring Plan for Phase II Remedial Action, Operable Unit 1, Naval Air Station Pensacola, Pensacola, Florida*. Bechtel Environmental, Inc.; Oak Ridge, Tennessee. (1999).
- Ecology and Environment, Inc. Contamination Assessment/Remedial Activities Investigation, Sanitary Landfill (Site 1), Naval Air Station Pensacola, Pensacola, Florida, Interim Data Report. E&E; Pensacola, Florida. (1991).
- EnSafe/Allen & Hoshall. Final Remedial Investigation Report for Site 1, Naval Air Station Pensacola, Pensacola, Florida. EnSafe/Allen & Hoshall; Memphis, Tennessee. (1996).
- EnSafe Inc. Proposed Plan for Site 1, Naval Air Station Pensacola, Florida. EnSafe; Memphis, Tennessee. (1997).
- Record of Decision for Site 1, Naval Air Station Pensacola, Florida. EnSafe; Memphis, Tennessee. (1998).
 - Final Remedial Design for Site 1, Naval Air Station Pensacola, Florida. EnSafe; Memphis, Tennessee. (1999).
 - Final Remedial Investigation Report Site 41 — Operable Unit 16 — NAS Pensacola Wetlands, Naval Air Station Pensacola, Florida. EnSafe; Memphis, Tennessee. (2007).
- Florida Department of Environmental Protection. Petroleum Contamination Site Cleanup Criteria, Chapter 62-770 Florida Administrative Code.
- Geraghty & Miller Inc. Verification Study: Assessment of Potential Groundwater Pollution at Naval Air Station Pensacola, Pensacola Florida. Geraghty & Miller Inc.; Tampa, Florida. (1984).
- Naval Facilities Engineering Command. Final Environmental Restoration Recordkeeping Manual. Naval Facilities Engineering Command; Washington, DC. (2009).



- Naval Energy and Environmental Support Activity. Initial Assessment Study of Naval Air Station Pensacola, Pensacola, Florida. NEESA; Port Hueneme, California. (1983).
- Northwest Florida Water Management District. Conceptual Model of the Sand and Gravel Aquifer Escambia County, Florida. NFWFMD; Pensacola, Florida. (1991).
- Resolution Consultants. November 2012 Groundwater Sampling Event, Operable Unit 1, Site 1, Sanitary Landfill, Naval Air Station Pensacola, Pensacola, Florida. (2013).
- Southeastern Geological Society. Florida Hydrogeological Units: Southeastern Geological Society Ad Hoc Committee on Florida Hydrostratigraphic Unit Definition, Florida Geologic Survey, Special Publication #28. (1986).
- Tetra Tech NUS, Inc. 3rd Annual Monitoring Report for Long-Term Monitoring for Operable Unit 1, Naval Air Station, Pensacola, Florida. TtNUS; Pittsburgh, Pennsylvania. (2003).
- Five-Year Review, Operable Units 1, 4, 10, 11, and 13, Naval Air Station Pensacola, Pensacola, Florida. TtNUS; Pittsburgh, Pennsylvania. (2008).
 - Optimization Study Report for Operable Unit 1 at Naval Air Station Pensacola. TtNUS; Pittsburgh, Pennsylvania. (April 2008).
 - 5th Annual Monitoring Report for Operable Unit 1 — Final Naval Air Station Pensacola, Florida. Tetra Tech; Pittsburgh, Pennsylvania. (January 2008).
 - Technical Memorandum, Reconnaissance Phase Flow Control Pilot Study for Operable Unit 1, Naval Air Station Pensacola, Pensacola Florida. Tetra Tech; Pittsburgh, Pennsylvania. (June 2009).
 - Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) for Long-term Groundwater Monitoring Operable Unit 1 — Site 1 Sanitary Landfill, Naval Air Station Pensacola, Florida. Tetra Tech; Pittsburgh, Pennsylvania. (July 2010).
 - (*in press*). Annual Groundwater Monitoring Report for Long-Term Monitoring, Operable Unit 1, Naval Air Station Pensacola, Pensacola, Florida. Tetra Tech; Pittsburgh, Pennsylvania. (2011)



- U.S. Department of Defense. *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 4.2. (2010).
- U.S. Environmental Protection Agency. *Test Methods for Evaluating Solid Waste; Physical/Chemical Methods (SW-846), 3rd Edition (including Update III)*. Office of Solid Waste and Emergency Response; Washington, DC. (1986).
- Publication 9203.1-02I, SACM Bulletins, Presumptive Remedies for Municipal Landfill Sites, April 1992, Vol. 1, #1, and February 1993, Vol. 2, #1, and SACM Bulletin Presumptive Remedies, August 1992, Vol. 1, #3.
 - *Guidance for Quality Assurance Project Plans, EPA QA/G-5*. EPA/240/R-02/009. Washington, DC. (2002).
 - *Uniform Federal Policy for Quality Assurance Project Plans. Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs, Parts 1, 2A, 2B, and 2C*. EPA-505-B-04-900A, Final, Version 1. (2005).
 - Field Branches Quality System and Technical Procedures. Retrieved from: <http://www.epa.gov/region4/sesd/fbqstp/> on 21 October 2013. .
 - U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, Office of Emergency and Remedial Response, Washington, District of Columbia. (2008).
 - *U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*. Office of Solid Waste and Emergency Response. (2010).
- University of Florida. Technical Report: Development of Cleanup Target Levels (CTLs) for Chapter 62-777, F.A.C. Center for Environmental & Human Toxicology, Gainesville, Florida. 26 February 2005.
- Wilkins, K.T., J.R. Wagner, and T. W. Allen. Hydrogeologic Data for the Sand-and-Gravel Aquifer in Southern Escambia County, Florida, Northwest Florida Water Management District, Technical File Report 85-2. (1985).

Appendix A
DQO Meeting Minutes

**NAS PENSACOLA
DATA QUALITY OBJECTIVES PLANNING MEETING
MAY 14, 2009**

Attendees:

- Greg Fraley (EPA R4) – EPA RPM
- Tracie Bolanos (FDEP) – State RPM
- Greg Campbell (NASP PWD) – Base Public Works representative
- Patty Marajh-Whittemore (NAVFAC RPM) – NAVFAC Project Manager
- Mike Singletary (NAVFAC) – NAVFAC Tech Support
- John Tucker (NAVFAC Navy Chemist) – NAVFAC Navy Chemist
- Gerry Walker (TtNUS) – TtNUS RPM
- Tom Johnston (TtNUS) – DQO Facilitator
- Katie Newman (TtNUS) - scribe

Supporting Documents: Multiple documents, including ROD - see draft UFP-SAP

Existing Data: See draft UFP-SAP data summaries (will be summarized in CSM presentation)

Opening Remarks (Johnston):

- Structured planning process and SAP are different. Planning outputs are translated into the SAP. The SAP must include bases/rationales for the plan as well as details of the plan that describe how to do the work and interpret the data.
- Should be able to progress rapidly through the planning for this site because there is a lot of information to help focus the effort and there has been general agreement on CSM and overall approach.
- Recapping current project status, CSM, etc. is intended to help focus the group. Please be patient and please participate so that when the meeting is concluded the agreements/consensus items achieved will serve as a strong foundation for completing the SAP.
- Assumptions will be documented to serve as a basis for the plan documented in the SAP.
- Action items (scope, steward, completion date) will be documented and distributed as soon as possible after the meeting.
- Overall planning objective is to develop an exit strategy to which all participants can agree to.

DQO Step 1 State the Problem

Conceptual Site Model, CSM (presented by G. Walker). Web presentation.

Site was open from 1950-1976 and it is approximately 80 acres in size with elevations between 8-20 feet above mean sea level (amsl). The site is covered with pine trees and scrub brush. The wastes on site include transformer oil, paint chips, air cylinders, and other various items of industrial waste.

Action Item #1 - T. Johnston- will send EPA Presumptive Remedy guidance to project team.
(Completed 5/14/09)

Figure 2-1 (shown in presentation) shows general area of primary landfill, monitoring well locations, and groundwater interceptor trench. The trench's purpose was to head off groundwater before it discharged into Wetland 3 to reduce iron concentrations coming from the landfill. It was shut down in 2008 because it was ineffective. The regulators (T. Bolanos and G. Fraley) discussed the point of compliance and both agreed it would be where the surface water sampling location is proposed to be located (green dot shown in the Technical Memorandum for Reconnaissance Phase Flow Control Pilot Study [Tech Memo] on Figure 11). It is located as close as you can get to Bayou Grande water body but staying far away from Wetland 3. T.

Bolanos said stated that she researched the wetland issue regarding proximity of a wetland to a landfill. FDEP position is that if a wetland is adjacent to a Superfund site, it is part of the potential contaminant source. Wetland 3 falls into this category. FDEP has developed a rationale for this.

Action Item #2 - T. Bolanos- Provide a copy of FDEP's rationale and get Concurrence Letter for the Tech Memo by the end of May 2009.

The groundwater at the site has a rating of G1, which is the highest rating for groundwater in Florida and it could be a drinking water source. Groundwater flow is radial to west/northwest, north, and east with predominant flow to north and northeast. The unit has been divided into three distinct zones: the surficial zone, the lower permeability zone, and the main producing zone. For portable water, NAS Pensacola relies on main producing zone wells at Corry Field, approximately three miles to the north. Site 1 investigations have been limited to the Sand-and-Gravel aquifer. Borings have been completed in the surficial zone, low permeability zone, and the upper portion of the main producing zone.

Action Item #3 - G. Walker will verify no deep water contamination and will ensure that this is described in the SAP.

Current Groundwater Conditions:

- Anoxic groundwater (showing limited aerobic degradation for chlorobenzene and VC) passes through a mixing zone at an aquifer-surface water body interface, the geochemistry changes naturally to an oxygen rich environment. The aerobic microbial population present in this zone (including streambed sediments) has been shown to create rapid mineralization of these compounds resulting in degradation to innocuous compounds
- Dilution and volatilization assist in the reduction of contaminants at the aquifer/surface water interface.
- As the aquifer approaches the surface water body, physical mixing occurs that results in dilution. This mixing occurs at most interfaces, but is enhanced where the aquifer discharges into a tidally influenced surface water body. Tidal pumping at the aquifer/surface water interface created by the change in tide will both bring a cyclic yet consistent source of oxygen and will continuously dilute the concentrations in this mixing zone. With this source of oxygen the volatile organic compounds (e.g., VC, benzene, and xylenes) will volatilize and will therefore be reduced in concentration.
- Based upon the existing contamination and geochemistry data sets and monitoring well network, aerobic oxidation of the organic constituents (VC, benzene, and xylene) cannot be definitively concluded as occurring at the site at this time.
- Contaminants persist in the intermediate interval wells. Where oxidizing conditions are present in either the shallow and/or intermediate intervals, there is little evidence of elevated contamination. For example, VC and benzene concentrations are persistent in well 01GI41 that is under reducing conditions, but the shallow well in the same cluster (01GS42) has oxidizing conditions and non detect levels of these contaminants.
- Based upon data collected through 2002, the 3rd Annual Groundwater Monitoring Report for OU1 recommended that monitoring only be continued for benzene, xylenes, VC, aluminum, cadmium, iron, and manganese. The remaining contaminants no longer exceeded applicable standards and were dropped from the LTMP.
- Impacts to sediment and soil are not a concern for this project (it has already been demonstrated that sediment is not impacted). Project team needs to make sure rationale behind this is included in the SAP.

Problem Definition:

- ROD states that performance standards (MCLs for 4 contaminants) must be satisfied for GW.

- In addition, chemical concentrations must satisfy GCTLs for an expanded list of chemicals to determine whether the remedy is protective of human health.
- Meet a criterion yet to be determined for iron in Bayou Grande to determine whether the remedy is protective of human health and the environment.

DQO Step 2: Decision Statements:

- Determine whether contaminant concentration in groundwater exceed MCLs for the 4 ROD contaminants. If they don't for the two most recent consecutive rounds of sampling, the planning team will convene to optimize the monitoring strategy (reduce sampling frequency, reduce number of wells, reduce number of contaminants). Team needs to determine whether 2 rounds or more would be needed to demonstrate – check guidance. If they do, continue monitoring and maintain LUCs and periodically evaluate the monitoring strategy to determine whether it can be optimized.

Action Item # 4 - T. Johnston/G. Walker- Research how many rounds of clean sampling is needed to determine how to optimize the monitoring strategy. Return results to team by May 25.

- Determine whether chemical concentrations in groundwater (chemicals that exceeded GCTLs in last two sampling rounds) exceed GCTLs. If they don't for the two most recent consecutive rounds of sampling, the planning team will convene to optimize the monitoring strategy (reduce sampling frequency, reduce number of wells, reduce number of contaminants). Team needs to determine whether 2 rounds or more would be needed to demonstrate – check guidance. If they do, continue monitoring and maintain LUCs and periodically evaluate the monitoring strategy to determine whether it can be optimized.
- Determine whether chemical concentrations in GW (chemicals that exceeded GCTLs in last two sampling rounds) have increased significantly. If they haven't, do not change the monitoring program; otherwise reconvene the project team to plan a course of action to respond to the observed increase.

Action Item #5 - T. Johnston: Prepare flow chart of decision logic and work with J. Tucker then send to team.

- Determine whether iron concentrations in SW exceed the criterion yet to be determined at the point of compliance. If they do exceed at the point of compliance, reconvene the project team to evaluate and select a remedial action.

Action Item #6 - TtNUS prepare straw man and distribute to team for review/comment.

DQO Step 3: Decision Inputs

Tech memo SW criteria

Total Xylenes
Total Iron

Action Items

Action Item No.	Responsible Party	Status	Due Date	Action Item
1	T. Johnston	Completed 5/14/09		Send EPA Presumptive Remedy Guidance to project team.
2	T. Bolanos	Working	5/31/09	Provide a copy of FDEP's rationale and get Concurrence Letter for the Tech Memo.
3	G. Walker	Working		Verify no deep water contamination and will ensure that this is described in the SAP.
4	T. Johnston/G. Walker	Working	5/25/09	Research how many rounds of clean sampling is needed to determine how to optimize the monitoring strategy.
5	T. Johnston	Working		Prepare flow chart of decision logic and work with J. Tucker then send to team
6	TtNUS	Working		Prepare straw man and distribute to team for review/comment.

**NAS PENSACOLA
CONTINUATION OF DATA QUALITY OBJECTIVES PLANNING MEETING
MAY 20, 2009**

Attendees:

- Patty Whittemore (NAVFAC RPM) – NAVFAC Proj. Mgr.
- Mike Singletary (NAVFAC) – NAVFAC Tech Support
- John Tucker (NAVFAC Gov. Chemist) – NAVFAC QA
- Gerry Walker (TtNUS) – Project Mgr
- Tom Johnston (TtNUS) – DQO Facilitator
- Katie Newman (TtNUS) - scribe

Tom recapped the first meeting, including the problem definition.

Tom stated that from his review of the guidance (specifically, RCRA Subpart F) it seemed that the best way to demonstrate compliance with the decision statement would be to provide statistical trend data to show that they are meeting MCLs, not just the two rounds of sampling. Tom asked the rest of the meeting attendees for their thoughts on including a statistical trend analysis.

Further discussion on the matter determined that the FDEP regulations only require 2 consecutive rounds of non-detects.

Tom then asked for clarification concerning the cap in place.

Gerry explained this was a non-engineered cap. There was reportedly a minimum of 2 feet of soil, on top of which pine trees had been planted.

Tom noted that the regulations seem to be written for an engineered cap, and that monitoring would need to take place as long as the landfill existed, since there is always a possibility that the cap could be breached. He noted that this makes it seem as though you would have to monitor forever, if there is no plan to remove the landfill contents. Tom noted that he thought that for the purposes of the exit strategy, wording should be changed to specify that as long as the landfill waste is in place, monitoring must continue.

The team discussed this further and noted that if included, this would need to be carefully worded, so that it did not appear to suggest that it was committing the Navy to removing the landfill.

Tom noted that his concern was that the conditions for how long sampling must occur are clearly stated.

Further discussion covered the topic of whether or not this applied only to the 4 ROD contaminants, or if it also included the expanded list of contaminants included in the PP.

Patty noted that the ROD was the legal binding document and that from her perspective, once the 4 ROD contaminants meet the MCLs then the requirements have been met and monitoring period is considered completed.

Tom noted that if they were associated with the landfill then it would seem that we'd have to monitor for all of those contaminants. Tom noted that he wanted to understand whether or not the expanded list of contaminants belong in the monitoring program, since they were not included in the ROD.

The point was made that if the decision was made not to monitor for the expanded list of contaminants, then this would have to be justified.

Tom noted that the 4 ROD contaminants are compared to the MCLs, but that the expanded contaminant list is compared to the GCTLs.

Table from Optimization Study- Gerry explained bold indicated contaminants that always had to be sampled for.

Suggestion to try to remove ROD Contaminants- but that would take a modification to the ROD.- because of landfill you would still have to monitor. Minimum that you would ever do is monitor 4 ROD contaminants, maybe once every 5 year to meet the 5 year review.

Two consecutive rounds of clean sampling are sufficient, tell that to regulators to get them to agree.

Monitor upstream, but that is not point of comp. We need point of compliance (poc) to keep from taking more action.

If we were monitoring upstream, if you saw an increase, no action would take place, , you would look for trends, but the only place you take action is the poc. We need additional data, so we take samples from other areas.

Intermediate points would serve some sort of purpose, but not sure how to put it in a decision statement. If we put an action limit in there, we have to be concerned with what to do with it if there is something detected. Need to have some monitoring system in place to warn you of exceedances. Up stream exceed some limit there, increase monitoring at poc, or put in temporary points between upstream points and poc to determine if it is increasing or seasonally driven.

Right now semi-annual sampling for gw and sw. Start out with annual now. Work into a logic that would give opportunity to make recommendations on how to optimize. Do on a sampling event basis- make an annual report. Reference Navy Monitoring Optimization Guide- make sure site doesn't become static. Plan should be able to change. Once a LTM plan is in place, should be reviewed/updated every year or so.

Step 3-

Action levels-

SW- not sure what the action levels are

Tech Memo- Tracie said she would send them to UF, but FDEP doesn't have funding for that, so she is going to review it, so hopefully in a month it will be back.

GeoChem Parameters? To support MNA in GW.

Water level measurements

SW Gauges

Step 4-

Temporal Boundaries- Any particular time it is best to sample?- POC is tidally influenced, so it is good to do it during the same tidal sequence. When tide is going into the bayou- sample on a falling tide. More important to do it on a flat tide? Because it would be easier to hit the exact point, so there is no inflow or outflow. Make sure it is not a rising tide. Do not need a minimum time between sample collection.

Appendix B
Field Standard Operating Procedures

Utility Clearance

Procedure 3-01

1.0 Purpose and Scope

- 1.1 This standard operating procedure (SOP) describes the process for determining the presence of subsurface utilities and other cultural features at locations where planned site activities involve the physical disturbance of subsurface materials.
- 1.2 This procedure is the Program-approved professional guidance for work performed by Resolution Consultants under the Comprehensive Long-Term Environmental Action Navy (CLEAN) contract (Contract Number N62470-11-D-8013).
- 1.3 The procedure applies to the following activities: soil gas surveying, excavating, trenching, drilling of borings and installation of monitoring and extraction wells, use of soil recovery or slide-hammer hand augers, and all other intrusive sampling activities.
- 1.4 The primary purpose of the procedure is to minimize the potential for damage to underground utilities and other subsurface features, which could result in physical injury, disruption of utility service, or disturbance of other subsurface cultural features.
- 1.5 If there are procedures, whether it be from Resolution Consultants, state, and/or federal, that are not addressed in this SOP and are applicable to utility clearance, those procedures should be added as an appendix to the project specific SAP.
- 1.6 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

- 2.1 Field and subcontractor personnel shall adhere to a site-specific health and safety plan (HASP).

3.0 Terms and Definitions

3.1 Utility

For the purposes of this SOP, a utility is defined as a manmade underground line or conduit, cable, pipe, vault or tank that is, or was, used for the transmission of material or energy (e.g., gas, electrical, telephone, steam, water or sewage, product transfer lines, or underground storage tanks).

3.2 As-Built Plans

As-built plans are plans or blueprints depicting the locations of structures and associated utilities on a property.

3.3 One-Call

The Utility Notification Center is the one-call agency for nationwide call before you dig. The Utility Notification Center is open 24 hours a day, and accepts calls from anyone planning to dig. The phone number 811 is the designated call before you dig phone number that directly connects you to your local one-call center. Additional information can be found at www.call811.com.

Calling before you dig ensures that any publicly owned underground lines will be marked so that you can dig around them safely. Having the utility lines marked not only prevents accidental damage to the lines, but prevents property damage and personal injuries that could result in breaking a line.

The following information will need to be provided when a call is placed to One-Call:

- Your name, phone number, company name (if applicable), and mailing address.
- What type of work is being done.
- Who the work is being done for.
- The county and city the work is taking place in.
- The address or the street where the work is taking place.
- Marking instructions, (specific instructions as to where the work is taking place).

Under normal circumstances it takes between 2 to 5 days from the time you call (not counting weekends or holidays) to have the underground lines marked. Because these laws vary from state to state, exactly how long it will take depends on where your worksite is located. You will be given an exact start time and date when your locate request is completed, which will comply with the laws in your area.

In the event of an emergency (any situation causing damage to life or property, or a service outage), lines can be marked sooner than the original given time if requested.

3.4 Toning

Toning is the process of surveying an area utilizing one or more surface geophysical methods to determine the presence or absence of underground utilities. Typically, toning is conducted after identifying the general location of utilities and carefully examining all available site utility plans. Each location is marked according to the type of utility being identified. In addition, areas cleared by toning are flagged or staked to indicate that all identified utilities in a given area have been toned.

4.0 Training and Qualifications

- 4.1 The **Contract Task Order (CTO) Manager** is responsible for verifying that these utility locating procedures are performed prior to the initiation of active subsurface exploration.
- 4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.
- 4.3 The **Field Manager** is responsible for ensuring that all utility locating activities are performed in accordance with this procedure.
- 4.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.0 Equipment and Supplies

- 5.1 Equipment and supplies necessary for locating subsurface utilities will be provided by the subcontractor; however, the project **Field Manager/Field Personnel** will provide any additional equipment and supplies as needed as well as maintain information regarding the utility clearance activities in the field logbook.

6.0 Procedure

Proceed with the following steps where subsurface exploration will include excavations, drilling, or any other subsurface investigative method that could damage utilities at a site. In addition to the steps outlined below, always exercise caution while conducting subsurface exploratory work.

6.1 **Prepare Preliminary Site Plan**

- Prepare a preliminary, scaled site plan depicting the proposed exploratory locations as part of the project specific Sampling and Analysis Plan (SAP) or Work Plan. Include as many of the cultural and natural features as practical in this plan.

6.2 **Review Background Information**

- Search existing plan files to review the as-built plans to identify the known location of utilities at the site. Plot the locations of utilities identified onto a preliminary, scaled site plan. Inform the CTO Manager if utilities lie within close proximity to a proposed exploration or excavation location. The CTO Manager will determine if it is necessary to relocate proposed sampling or excavation locations.
- Include the utility location information gathered during previous investigations (e.g., remedial investigation or remedial site evaluation) in the project design documents for removal or remedial actions. In this manner, information regarding utility locations collected during implementation of a CTO can be shared with the subcontractor during implementation of a particular task order. In many instances, this will help to reduce the amount of additional geophysical surveying work the subcontractor may have to perform.
- Conduct interviews with onsite and facility personnel familiar with the site to obtain additional information regarding the known and suspected locations of underground utilities. In addition, if appropriate, contact shall be made with local utility companies to request their help in locating underground lines. Pencil in the dimensions, orientation, and depth of utilities, other than those identified on the as-built plans, at their approximate locations on the preliminary plans. Enter the type of utility, the personnel who provided the information, and the date the information was provided into the field log.
- During the pre-field work interviewing process, the interviewer will determine which site personnel should be notified in the event of an incident involving damage to existing utilities. Record this information in the field logbook with the corresponding telephone numbers and addresses.

6.3 **Site Visit/Locate Utilities/Toning**

- Prior to the initiation of field activities, the Field Task Manager or similarly qualified field personnel shall visit the site and note existing structures and evidence of associated utilities, such as fire hydrants, irrigation systems, manhole and vault box covers, standpipes, telephone switch boxes, free-standing light poles, gas or electric meters, pavement cuts, and linear depression. Compare notes of the actual site configuration to the preliminary site plan. Note deviations in the field logbook and on the preliminary site plan. Accurately locate or survey and clearly mark with stakes, pins, flags, paint, or other suitable devices all areas where subsurface exploration is proposed. These areas shall correspond with the locations drawn on the preliminary site plan.
- Following the initial site visit by the Field Task Manager, a trained utility locating subcontractor will locate, identify, and tone all utilities depicted on the preliminary site plan. The Field Task Manager or similarly qualified field personnel shall visit the site and identify the areas of subsurface disturbance with white spray paint, chalk, white pin flags or some other easily identifiable marking. The utility locator should utilize appropriate sensing equipment to attempt to locate utilities that might not have appeared on the as-built plans. At a minimum, the utility subcontractor should utilize a metal detector and/or magnetometer; however, it is important to consider the possibility that non-metallic utilities or tanks might be present at the site. Use other appropriate surface geophysical methods such as Ground Penetrating Radar, Radiodetection, etc. as appropriate. Clear proposed exploration areas of all utilities in the immediate area where subsurface exploration is proposed. Clearly tone all anomalous areas. Clearly identify all toned areas on the preliminary site plan. All utilities near the area of subsurface disturbance should also be marked out by the utility subcontractor using the universal colors for subsurface utilities (i.e., red – electric; blue – water; green – sewer; yellow – gas; etc.). After toning the site and plotting all known or suspected buried utilities on the preliminary site plan, the utility locator shall provide the Field Task Manager with a copy of the completed preliminary

site plan. Alternatively, the Field Task Manager or designee shall document the results of the survey on the preliminary site plan.

- Report to the Field Task Manager anomalous areas detected and toned that are in close proximity to the exploration or excavation areas. The Field Task Manager shall determine the safe distance to maintain from the known or suspected utility. It may be necessary to relocate the proposed exploration or excavation areas. If this is required, the Field Task Manager or designee shall relocate them and clearly mark them using the methods described above. Completely remove the markings at the prior location. Plot the new locations on the site plan and delete the prior locations from the plan. In some instances, such as in areas extremely congested with subsurface utilities, it may be necessary to dig by hand or use techniques such as air knife to determine the location of the utilities.

6.4 **Prepare Site Plan**

- Prior to the initiation of field activities, draft a final site plan that indicates the location of subsurface exploration areas and all known or suspected utilities present at the site. Provide copies of this site plan to the Navy Technical Representative (NTR), the CTO Manager, and the subcontractor who is to conduct the subsurface exploration/excavation work. Review the site plan with the NTR to verify its accuracy prior to initiating subsurface sampling activities.

7.0 **Quality Control and Assurance**

- 7.1 Utility locating must incorporate quality control measures to ensure conformance to these and the project requirements.

8.0 **Records, Data Analysis, Calculations**

- 8.1 A bound field logbook will be kept detailing all activities conducted during the utility locating procedure.
- 8.2 The logbook will describe any changes and modifications made to the original exploration plan. The trained utility locator shall prepare a report and keep it in the project file. Also, a copy of the final site plan will be kept in the project file.

9.0 **Attachments or References**

Department of Defense, United States (DoD). 2005. [Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual](http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf). Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Author	Reviewer	Revisions (Technical or Editorial)
Caryn DeJesus Senior Scientist	Bob Shoemaker Senior Scientist	Rev 0 – Initial Issue (June 2012)

Logbooks

Procedure 3-02

1.0 Purpose and Scope

- 1.1 This standard operating procedure (SOP) describes the activities and responsibilities pertaining to the identification, use, and control of logbooks and associated field data records.
- 1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

- 2.1 In order to keep the logbook clean, store it in a clean location and use it only when outer gloves used for PPE have been removed.

3.0 Terms and Definitions

3.1 Logbook

A logbook is a bound field notebook with consecutively numbered, water-repellent pages that is clearly identified with the name of the relevant activity, the person assigned responsibility for maintenance of the logbook, and the beginning and ending dates of the entries.

3.2 Data Form

A data form is a predetermined format utilized for recording field data that may become, by reference, a part of the logbook (e.g., soil boring logs, trenching logs, surface soil sampling logs, groundwater sample logs, and well construction logs are data forms).

4.0 Training and Qualifications

- 4.1 The **Contract Task Order (CTO) Manager** or **designee** is responsible for determining which team members shall record information in field logbooks and for obtaining and maintaining control of the required logbooks. The **CTO Manager** shall review the field logbook on at least a monthly basis. The **CTO Manager** or **designee** is responsible for reviewing logbook entries to determine compliance with this procedure and to ensure that the entries meet the project requirements.
- 4.2 A knowledgeable individual such as the **Field Manager**, **CTO Manager**, or **Program Quality Manager** shall perform a technical review of each logbook at a frequency commensurate with the level of activity (weekly is suggested, or, at a minimum, monthly). Document these reviews by the dated signature of the reviewer on the last page or page immediately following the material reviewed.
- 4.3 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.
- 4.4 The **Field Manager** is responsible for ensuring that all **field personnel** follow these procedures and that the logbook is completed properly and daily. The **Field Manager** is also responsible for submitting copies to the **CTO Manager**, who is responsible for filing them and submitting a copy (if required by the CTO Statement of Work).
- 4.5 The **logbook user** is responsible for recording pertinent data into the logbook to satisfy project requirements and for attesting to the accuracy of the entries by dated signature. The **logbook user** is also responsible for safeguarding the logbook while having custody of it.

4.6 All **field personnel** are responsible for the implementation of this procedure.

5.0 Equipment and Supplies

5.1 Field logbooks shall be bound field notebooks with water-repellent pages.

5.2 Pens shall have indelible black ink.

6.0 Procedure

6.1 The field logbook serves as the primary record of field activities. Make entries chronologically and in sufficient detail to allow the writer or a knowledgeable reviewer to reconstruct the applicable events. Store the logbook in a clean location and use it only when outer gloves used for personal protective equipment (PPE) have been removed.

6.2 Individual data forms may be generated to provide systematic data collection documentation. Entries on these forms shall meet the same requirements as entries in the logbook and shall be referenced in the applicable logbook entry. Individual data forms shall reference the applicable logbook and page number. At a minimum, include names of all samples collected in the logbook even if they are recorded elsewhere.

6.3 Enter field descriptions and observations into the logbook, as described in Attachment 1, using indelible black ink.

6.4 Typical information to be entered includes the following:

- Dates (month/day/year) and times (military) of all on-site activities and entries made in logbooks/forms;
- Site name and description;
- Site location by longitude and latitude, if known;
- Weather conditions, including temperature and relative humidity;
- Fieldwork documentation, including site entry and exit times;
- Descriptions of, and rationale for, approved deviations from the work plan (WP) or field sampling plan;
- Field instrumentation readings;
- Names, job functions, and organizational affiliations of on-site personnel;
- Photograph references;
- Site sketches and diagrams made on site;
- Identification and description of sample morphology, collection locations, and sample numbers;
- Sample collection information, including dates (month/day/year) and times (military) of sample collections, sample collection methods and devices, station location numbers, sample collection depths/heights, sample preservation information, sample pH (if applicable), analysis requested (analytical groups), etc., as well as chain-of-custody (COC) information such as sample identification numbers cross-referenced to COC sample numbers;
- Sample naming convention;
- Field quality control (QC) sample information;
- Site observations, field descriptions, equipment used, and field activities accomplished to reconstruct field operations;

- Meeting information;
 - Important times and dates of telephone conversations, correspondence, or deliverables;
 - Field calculations;
 - PPE level;
 - Calibration records;
 - Contractor and subcontractor information (address, names of personnel, job functions, organizational affiliations, contract number, contract name, and work assignment number);
 - Equipment decontamination procedures and effectiveness;
 - Laboratories receiving samples and shipping information, such as carrier, shipment time, number of sample containers shipped, and analyses requested; and
 - User signatures.
- 6.5 The logbook shall reference data maintained in other logs, forms, etc. Correct entry errors by drawing a single line through the incorrect entry, then initialing and dating this change. Enter an explanation for the correction if the correction is more than for a mistake.
- 6.6 At least at the end of each day, the person making the entry shall sign or initial each entry or group of entries.
- 6.7 Enter logbook page numbers on each page to facilitate identification of photocopies.
- 6.8 If a person's initials are used for identification, or if uncommon acronyms are used, identify these on a page at the beginning of the logbook.
- 6.9 At least weekly and preferably daily, the **preparer** shall photocopy and retain the pages completed during that session for backup. This will prevent loss of a large amount of information if the logbook is lost.

7.0 Quality Control and Assurance

- 7.1 Review per Section 4.2 shall be recorded.

8.0 Records, Data Analysis, Calculations

- 8.1 Retain the field logbook as a permanent project record. If a particular CTO requires submittal of photocopies of logbooks, perform this as required.
- 8.2 Deviations from this procedure shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

9.0 Attachments or References

- 9.1 Attachment 1 – Description of Logbook Entries
- 9.2 Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue

Attachment 1

Description of Logbook Entries

Logbook entries shall be consistent with Section A.1.4 *Field Documentation SOPs* of the UFP-QAPP Manual (DoD 2005) and contain the following information, as applicable, for each activity recorded. Some of these details may be entered on data forms, as described previously.

Name of Activity	For example, Asbestos Bulk Sampling, Charcoal Canister Sampling, Aquifer Testing.
Task Team Members and Equipment	Name all members on the field team involved in the specified activity. List equipment used by serial number or other unique identification, including calibration information.
Activity Location	Indicate location of sampling area as indicated in the field sampling plan.
Weather	Indicate general weather and precipitation conditions.
Level of PPE	Record the level of PPE (e.g., Level D).
Methods	Indicate method or procedure number employed for the activity.
Sample Numbers	Indicate the unique numbers associated with the physical samples. Identify QC samples.
Sample Type and Volume	Indicate the medium, container type, preservative, and the volume for each sample.
Time and Date	Record the time and date when the activity was performed (e.g., 0830/08/OCT/89). Use the 24-hour clock for recording the time and two digits for recording the day of the month and the year.
Analyses	Indicate the appropriate code for analyses to be performed on each sample, as specified in the WP.
Field Measurements	Indicate measurements and field instrument readings taken during the activity.
Chain of Custody and Distribution	Indicate chain-of-custody for each sample collected and indicate to whom the samples are transferred and the destination.
References	If appropriate, indicate references to other logs or forms, drawings, or photographs employed in the activity.
Narrative (including time and location)	Create a factual, chronological record of the team's activities throughout the day including the time and location of each activity. Include descriptions of general problems encountered and their resolution. Provide the names and affiliations of non-field team personnel who visit the site, request changes in activity, impact the work schedule, request information, or observe team activities. Record any visual or other observations relevant to the activity, the contamination source, or the sample itself. It should be emphasized that logbook entries are for recording data and chronologies of events. The logbook author must include observations and descriptive notations, taking care to be objective and recording no opinions or subjective comments unless appropriate.
Recorded by	Include the signature of the individual responsible for the entries contained in the logbook and referenced forms.
Checked by	Include the signature of the individual who performs the review of the completed entries.

Sample Labeling and Chain of Custody Procedures

Procedure 3-03A

1.0 Purpose and Scope

- 1.1 The purpose of this standard operating procedure is to establish standard protocols for all field personnel for use in maintaining field and sampling activity records, labeling samples, ensuring that proper sample custody procedures are utilized, and completing chain-of-custody/analytical request forms.
- 1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

Not applicable

3.0 Definitions

3.1 Logbook

A logbook is a bound field notebook with consecutively numbered, water-repellent pages that is clearly identified with the name of the relevant activity, the person responsible for maintenance of the logbook, and the beginning and ending dates of the entries.

3.2 Chain-of-Custody

Chain-of-custody (COC) is documentation of the process of custody control. Custody control includes possession of a sample from the time of its collection in the field to its receipt by the analytical laboratory, and through analysis and storage prior to disposal.

4.0 Training and Qualifications

- 4.1 The **CTO Manager**, or designee, is responsible for determining which team members shall record information in the field logbook and for checking sample logbooks and COC forms to ensure compliance with these procedures. The **CTO Manager**, or designee, shall review COC forms at the completion of each sampling event.
- 4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.
- 4.3 The **Field Manager** is responsible for ensuring that all field equipment is decontaminated according to this procedure.
- 4.4 The **Project Chemist**, or designee, is responsible for verifying that the COC/analytical request forms have been completed properly and match the sampling and analytical plan. The **Project Chemist**, or designee, is responsible for notifying the laboratory, data managers, and data validators in writing if analytical request changes are required as a corrective action. These small changes are different from change orders, which involve changes to the scope of the subcontract with the laboratory and must be made in accordance with a respective contract.
- 4.5 All **Field Personnel** are responsible for recording pertinent data onto the COC forms to satisfy project requirements and for attesting to the accuracy of the entries by dated signature.

5.0 Procedure

This procedure provides standards for labeling the samples, documenting sample custody, and completing COC/analytical request forms. The standards presented in this section shall be followed to ensure that samples collected are maintained for their intended purpose and that the conditions encountered during field activities are documented.

5.1 Sample Labeling

Affix a waterproof sample label with adhesive backing to each individual sample container. Record the following information with a waterproof marker on each label:

- Project name or number (optional)
- COC sample number
- Date and time of collection
- Sampler's initials
- Matrix (optional)
- Sample preservatives (if applicable)
- Analysis to be performed on sample (This shall be identified by the method number or name identified in the subcontract with the laboratory)

These labels may be obtained from the analytical laboratory or printed from a computer file onto adhesive labels.

5.2 Custody Procedures

For samples intended for chemical analysis, sample custody procedures shall be followed through collection, transfer, analysis, and disposal to ensure that the integrity of the samples is maintained. A description of sample custody procedures is provided below.

Sample Collection Custody Procedures

According to the EPA guidelines, a sample is considered to be in custody if one of the following conditions is met:

- It is in one's actual physical possession or view
- It is in one's physical possession and has not been tampered with (i.e., it is under lock or official seal)
- It is retained in a secured area with restricted access
- It is placed in a container and secured with an official seal such that the sample cannot be reached without breaking the seal

Place custody seals on shipping coolers (and sample jars, if required) if the cooler/container is to be removed from the sampler's custody. Place a minimum of two custody seals in such a manner that they must be broken to open the containers or coolers. Label the custody seals with the following information:

- Sampler's name or initials
- Date and time that the sample/cooler was sealed

These seals are designed to enable detection of sample tampering. An example of a custody seal is shown in Attachment 1.

Field personnel shall also log individual samples onto COC forms (carbon copy or computer generated) when a sample is collected. These forms may also serve as the request for analyses. Procedures for completing these forms are discussed in Section 0, indicating sample identification number, matrix, date and time of collection, number of containers, analytical methods to be performed on the sample, and preservatives added (if any). The samplers will also sign the COC form signifying that they were the personnel who collected the samples. The COC form shall accompany the samples from the field to the laboratory. When a cooler is ready for shipment to the analytical laboratory, the person delivering the samples for transport will sign and indicate the date and time on the accompanying COC form. One copy of the COC form will be retained by the sampler and the remaining copies of the COC form shall be placed inside a self-sealing bag and taped to the inside of the cooler. Each cooler must be associated with a unique COC form. Whenever a transfer of custody takes place, both parties shall sign and date the accompanying carbon copy COC forms, and the individual relinquishing the samples shall retain a copy of each form. One exception is when the samples are shipped; the delivery service personnel will not sign or receive a copy because they do not open the coolers. The laboratory shall attach copies of the completed COC forms to the reports containing the results of the analytical tests. An example COC form is provided in Attachment 2.

5.3 **Completing COC/Analytical Request Forms**

COC form/analytical request form completion procedures are crucial in properly transferring the custody and responsibility of samples from field personnel to the laboratory. This form is important for accurately and concisely requesting analyses for each sample; it is essentially a release order from the analysis subcontract.

Attachment 2 is an example of a completed COC/analytical request form that may be used by field personnel, with box numbers identified and discussed in text below. Multiple copies may be tailored to each project so that much of the information described below need not be handwritten each time. Each record on the form (Attachment 2) is identified with a bold number corresponding to the instructions given below.

1. Record the project name, site location.
2. Record the site location, including the state.
3. Record the Contract Task Order number
4. Record the Resolution Consultants Task Order Manager
5. Record the sampler/site phone or cell number (if applicable).
6. Record the laboratory name where the samples were sent.
7. Record the requested turnaround time, in days. If a specific turnaround time is required to meet project objectives, but was not indicated on the laboratory service request form submitted to the purchasing department, the sampler, project manager, or site manager should contact the purchasing department so the laboratory contract can be modified.
8. Record the COC number that is defined by the sampler and should be unique throughout the project's history. An example would be to use the sampler's initials followed by the data. If multiple custodies are generated on a given day, use a unique sequential identifier. Example: CRC040105A, CRC040105B
9. Record the purchase order number provided by the purchasing department.
10. Record the page and total number of COC forms used in a shipment.
11. Record the project, and phase applicable to the sampling task.
12. Record the two-character code corresponding to the *chemical* preservation type, which is found on the bottom of the COC form. If no chemical preservation was added to the sample, the field should be left blank. Temperature preservation need not be documented at this location, but will be indicated elsewhere on the COC form (see 33).

13. List the requested analysis. Whenever possible, list the corresponding analytical method. (e.g., VOCs, 8260).
14. For Lab identification use only.
15. Record the full unique sample identification as detailed in the Site's Sampling and Analysis Plan.
16. Record the location identification, which is a shortened ID used for presentation and mapping, as detailed in the Site's Sampling and Analysis Plan.
17. Record the sample date using the format mm/dd/yy.
18. Record the sample time using the military format of hhmm.
19. Record the matrix code of the sample, which is located at the bottom of the COC form. The matrix code is a crucial element of the Navy's data management system. For simplicity, only typical matrix codes are listed on the bottom COC form, but below is a complete listing of all applicable Navy matrix codes:

Table 1
Navy Matrix Codes

Matrix Code	Matrix Code Description	Matrix Code	Matrix Code Description
AA	Ambient air	RK	Rock
AC	Composite air sample	SB	Bentonite
AD	Air - Drilling	SBS	Sub-surface soil (> 6")
AIN	Integrated air sample (under sample form of gas)	SC	Cement/Concrete
AQ	Air quality control matrix	SD	Drill cuttings — solid matrix
AQS	Aqueous	SE	Sediment
ASB	Asbestos	SEEP	SEEP
ASBF	Asbestos-Fibrous	SF	Filter sand pack
ASBNF	Asbestos-Non-Fibrous	SJ	Sand
AVE	Air-Vapor extraction, effluent	SK	Asphalt
AX	Air sample from unknown origin	SL	Sludge
BK	Brick	SM	Water filter (solid material used to filter water)
BS	Brackish sediment	SN	Miscellaneous solid/building materials
CA	Cinder ash	SO	Soil
CK	Caulk	SP	Casing (PVC, stainless steel, cast iron, iron pipe)
CN	Container	SQ	Soil/Solid quality control matrix
CR	Carbon (usually for a remediation system)	SS	Scrapings
DF	Dust/Fallout	SSD	Subsurface sediment
DR	Debris/rubble	STKG	Stack gas
DS	Storm drain sediment	STPM	Stripper Tower Packing Media
DT	Trapped debris	SU	Surface soil (less than 6 inches)
EF	Emissions flux	SW	Swab or wipe
EW	Elutriate water	SZ	Wood
FB	Fibers	TA	Animal tissue
FL	Forest litter	TP	Plant tissue
GE	Soil gas effluent — stack gas (from system)	TQ	Tissue QC
GI	Soil gas influent (into system)	TX	Tissue
GL	Headspace of liquid sample	UNK	Unknown
GQ	Gaseous or Headspace QC	W	Water (not groundwater, unspecified)
GR	Gravel	WA	Drill cuttings - aqueous mix
GS	Soil gas	WB	Brackish Water

**Table 1
Navy Matrix Codes**

Matrix Code	Matrix Code Description	Matrix Code	Matrix Code Description
GT	Grit	WC	Drilling water (used for well construction)
IC	IDW Concrete	WD	Well development water
IDD	IDW Solid	WF	Freshwater (not groundwater)
IDS	IDW soil	WG	Ground water
IDW	IDW Water	WH	Equipment wash water
IW	Interstitial water	WI	Ground water influent (into system)
LA	Aqueous phase of a multiphase liquid/soil	WL	Leachate
LF	Product (floating or free)	WM	Marine water
LQ	Organic liquid quality control matrix	WN	Pore water
MA	Mastic	WO	Ocean water
MO	Mortar	WP	Drinking water
MR	Marine sediment	WQ	Water for QC samples
MS	Metal shavings	WR	Ground water effluent (from system)
NS	Near-surface soil	WS	Surface water
PA	Paper	WT	Composite groundwater sample
PC	Paint Chips	WU	Storm water
PP	Precipitate	WW	Waste water
RE	Residue		

Field QC blanks will require matrix codes that identify the type of blank associated with parent sample. Aqueous field QC blanks are not automatically identified with a matrix code of "WQ," indicating a water quality control blank; they are only identified with a matrix code of "WQ" if the associated samples are also aqueous. Trip blanks, field blanks, and equipment rinsate blanks collected in association with **soil** samples will be identified with a matrix code of "SQ," even though the actual matrix is aqueous, because the blanks were collected to assess potential contamination imparted during decontamination activities or transport of **soil** samples.

20. Record the sample type code, which is located at the bottom of the COC form. The sample type is a crucial element of the EQuIS data management system. For simplicity, only typical sample type codes are listed on the bottom of the COC form, but below is a list of all applicable Navy field sample type codes:

**Table 2
Navy Sample Type Codes**

Sample Type Code	Sample Type Code Description
AB	Ambient condition blank
BIOCON	Bioassay control sample
BS	Blank spike
BSD	Blank spike duplicate
EB	Equipment blank
EBD	Equipment blank/rinsate duplicate
FB	Field blank
FD	Field duplicate
FS	Field spike
IDW	Purge and rinsate water
LB	Lab Blank

Table 2
Navy Sample Type Codes

Sample Type Code	Sample Type Code Description
LR	Lab Replicate
MB	Material blank
MIS	Multi-Incremental Sample
MS	Matrix spike
N	Normal (Regular)
PE	Performance evaluation
PURGE	Purge water sample
RD	Regulatory duplicate
SB	Source blank
SBD	Source blank duplicate
SCREEN	Screening Sample
SD	Matrix spike duplicate
SPLIT	Sample split
SRM	Standard reference material
TB	Trip Blank
TBD	Trip blank duplicate
TBR	Trip blank replicate

Field duplicate samples — Field duplicates will be identified using the format detailed in the Site's Sampling and Analysis Plan. However, field duplicates will also be differentiated from the parent sample on the chain-of-custody form. The parent sample will have a sample type code of "N," for normal environmental sample; while its duplicate will have a sample type code of "FD."

21. Record whether the sample is field filtered with a "Y" or not field filtered with an "N." If a project requires collecting samples for both total and dissolved constituents, the same sample and location ID is used for both (see 15 and 16); however, the sampler will indicate whether the sample is field filtered at this location on the COC form. This field must always be filled out; even when soil samples are collected (where "N" appropriately applies, in most cases).
22. Record the total number of containers that are submitted for all of the tests. This must add up to the total number of containers listed for each individual test in 23.
23. Record the number of containers for each test. Do not use Xs, rather indicate the number of containers submitted for each test listed in 14. For example, Sample 010MW007002 requires analysis for VOCs (8260), and SVOCs (8270). Record 3 under the VOC analysis and 2 under the SVOC (assuming 3 containers were submitted for VOCs and 2 were submitted for SVOCs). The total number of containers in this example is 5, which should be the total number of containers listed in 22. Extra containers submitted for matrix spike/matrix spike duplicates (MS/MSDs) will be appropriately recorded.
24. Indicate if extra sample volume was included for MS/MSD analysis using an "X." Samples to be used for MS/MSDs will use the same sample ID and location ID (see 15 and 16), but will be collected in triplicate, particularly for liquid samples, to ensure the analytical laboratory receives sufficient volume for the analyses.
25. Indicate if the samples should be held by the laboratory for future testing using an "X."
26. Record any field comments.
27. Reserved for laboratory comments.

28. Indicate the total number of coolers in each shipment. *Note:* When multiple coolers are submitted, each should contain a COC form.
29. Signature(s) of the person(s) relinquishing sample custody.
30. Signature(s) of the person(s) receiving sample custody.
31. Indicate whether the samples are iced, by checking the appropriate response.
32. Indicate the method of shipment (e.g., FedEx, hand-delivered, laboratory courier).
33. Record the airbill number when a commercial courier is used. This is particularly important when multiple coolers are sent in the same shipment or when the laboratory is sent the COC form in advance of receiving samples because it aids in tracking lost coolers.
34. Record the date the coolers were shipped.

COC forms tailored to each CTO can be drafted and printed onto multiple forms. This eliminates the need to rewrite the analytical methods column headers each time. It also eliminates the need to write the project manager, name, and number; QC Level; turnaround time; and the same general comments each time.

Complete one COC form per cooler. Whenever possible, place all volatile organic analyte vials into one cooler in order to reduce the number of trip blanks. Complete all sections and be sure to sign and date the COC form. One copy of the COC form must remain with the field personnel.

6.0 Records

The COC/analytical request form shall be faxed or emailed approximately daily to the Project Chemist, or designee for verification of accuracy. Following the completion of sampling activities, the sample logbook and COC forms will be transmitted to the CTO Manager for storage in project files. The original COC/analytical request form shall be submitted by the laboratory along with the data delivered. Any changes to the analytical requests that are required shall be made in writing to the laboratory. A copy of this written change shall be sent to the data validators and placed in the project files. The reason for the change shall be included in the project files so that recurring problems can be easily identified.

7.0 References and Attachments

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/-fedfac/pdf/ufp_qapp_v1_0305.pdf.

Attachment 1: Chain-of-Custody Seal

Attachment 2: Generic Chain-of-Custody/Analytical Request Form

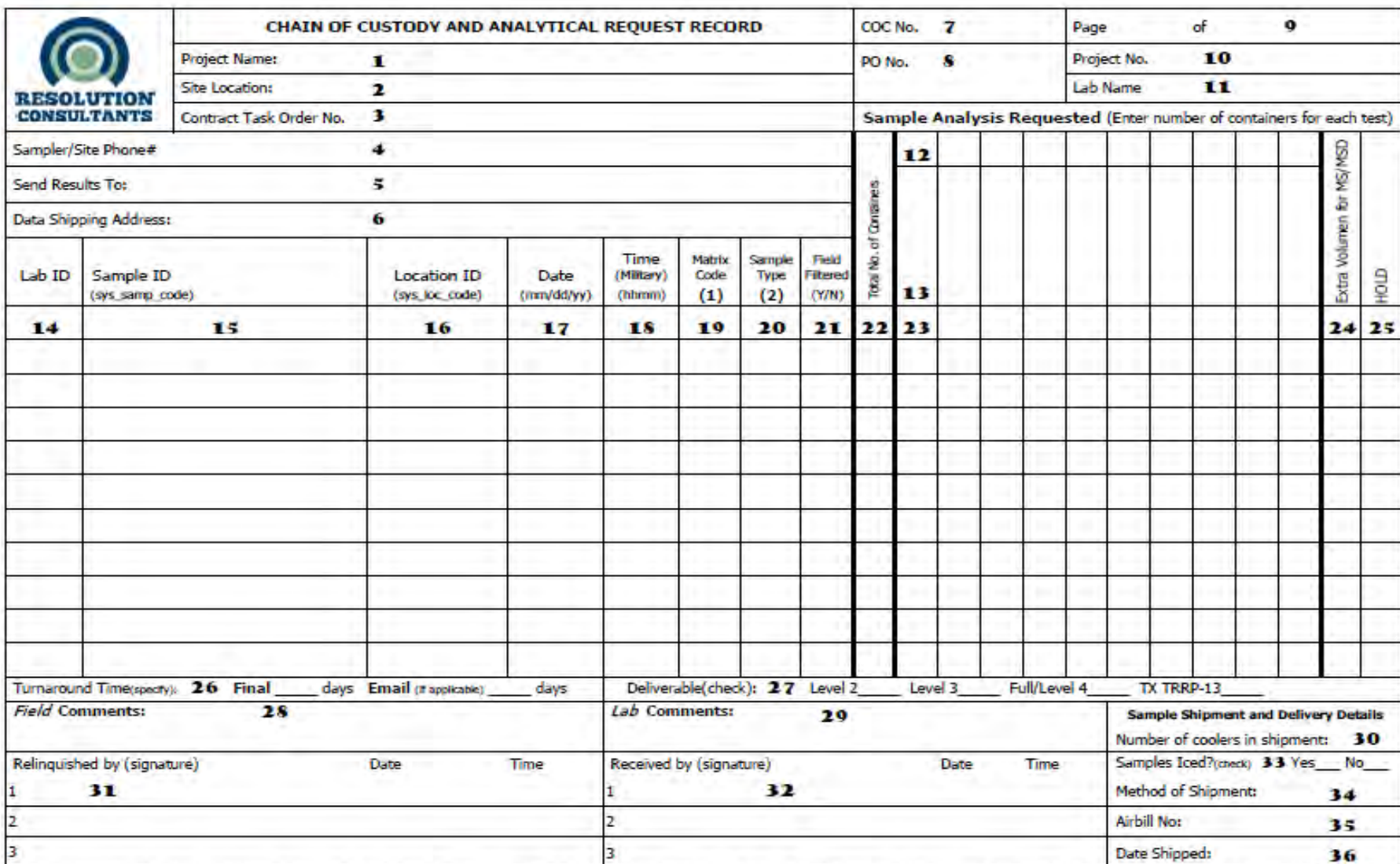
Author	Reviewer	Revisions (Technical or Editorial)
Tina Cantwell QA Officer	Ben Brantley Project Manager	Rev 0 — Initial Issue

Attachment 1
Chain-of-Custody Seal

EXAMPLE CHAIN-OF-CUSTODY SEAL

[LABORATORY]	SAMPLE NO.	DATE	SEAL BROKEN BY
	SIGNATURE		DATE
	PRINT NAME AND TITLE (<i>Inspector, Analyst or Technician</i>)		

Attachment 2
Example Chain-of-Custody/Analytical Request Form



(1) **AA**—Ambient air, **AQ**—Air quality control, **ASB**—Asbestos, **CK**—Caulk, **DS**—Storm drain sediment, **GS**—Sol gas, **IC**—IDW Concrete, **IDD**—IDW Solid, **IDG**—IDW soil, **IDW**—IDW Water, **LF**—Free Product, **MA**—Mastic, **PC**—Paint Chips, **SC**—Cement/Concrete, **SE**—Sediment, **SL**—Sludge, **SO**—Sol, **SQ**—Sol/Solid quality control, **SSD**—Subsurface sediment, **SU**—Surface soil (<6 in), **SW**—Sweep or wipe, **TA**—Animal tissue, **TP**—Plant tissue, **TQ**—Tissue quality control, **WG**—Ground water, **WL**—Leachate, **WO**—Ocean water, **WP**—Drinking water, **WQ**—Water quality control, **WR**—Ground water effluent, **WS**—Surface water, **WU**—Storm water, **WW**—Waste water

(2) **Sample Type:** **AB**—Ambient Bk, **EB**—Equipment Bk, **FB**—Field Bk, **FD**—Field Duplicate Sample, **IDW**—Investigative-Derived Water, **MIS**—Incremental Sampling Methodology, **N**—Normal Environmental Sample, **PE**—Performance Eval., **TR**—Trip Bk

(3) **Preservative added:** **HA**—Hydrochloric Acid, **NI**—Nitric Acid, **SH**—Sodium Hydroxide, **SA**—Sulfuric Acid, **AA**—Ascorbic Acid, **HX**—Hexene, **MS**—Methanol, **SS**—sodium bisulfate, **ST**—Sodium Thiosulfate. If **NO preservative** added leave blank

Sample Handling, Storage, and Shipping of Low Level Environmental Samples

Procedure 3-04A

1.0 Purpose and Scope

- 1.1 This Standard Operating Procedure (SOP) sets forth the methods for use by personnel engaged in handling, storing, and transporting low level environmental samples.
- 1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

- 2.1 To avoid lifting injuries associated with heavy coolers, use the large muscles of the legs, not the back. Use dollies if possible.
- 2.2 When using tools for cutting purposes, cut away from yourself. The use of appropriate, task specific cutting tools is recommended.
- 2.3 Wear proper gloves, such as blue nitrile and latex, as defined in the site-specific project health and safety plan, when handling sample containers to avoid contacting any materials that may have spilled out of the sample containers.

3.0 Terms and Definitions

DOT — Department of Transportation

4.0 Training and Qualifications

- 4.1 The **Contract Task Order (CTO) Manager** is responsible for verifying that these procedures are performed prior to the initiation of active subsurface exploration.
- 4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.
- 4.3 The **Field Manager** is responsible for ensuring that sample handling, storage, and shipping are performed in accordance with this procedure.
- 4.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.0 Procedures

5.1 Handling and Packaging

Environmental samples should be packaged prior to shipment using the following procedures:

- 1. Allow sufficient headspace in all bottles (except volatile organic analysis containers with a septum seal) to compensate for any pressure and temperature changes (approximately 1 percent of the volume of the container).
- 2. Ensure that the lids on all bottles are tight (will not leak).

3. Glass bottles should be wrapped in bubble wrap — preferably sealable bubble wrap sample bags, if available. Place bottles in separate and appropriately-sized polyethylene bags and seal the bags.
4. Select a sturdy cooler in good repair. Secure and tape the drain plug with fiber or duct tape inside and outside. Line the cooler with a large heavy-duty plastic bag.
5. Place cushioning/absorbent material in the bottom of the cooler, if available, and then place the containers in the cooler with sufficient space to allow for the addition of cushioning between the containers.
6. Put "blue ice" (or ice that has been "double bagged" in heavy-duty polyethylene bags and properly sealed) on top of and/or between the containers. Fill all remaining space between the containers with bubble wrap or other suitable absorbent material.
7. Securely fasten the top of the large garbage bag with packaging tape.
8. Place the completed Chain-of-Custody (COC) Record into a sealed plastic bag, and tape the bag to the inner side of the cooler lid.
9. Close the cooler and securely tape (preferably with fiber tape) the top of the cooler shut. COC seals should be affixed to opposing sides of the cooler within the securing tape so that the cooler cannot be opened without breaking the seal.

5.2 Shipping

Follow all appropriate DOT regulations (e.g., 49 Code of Federal Regulations, Parts 171-179) for shipment of air, soil, water, and other samples. Elements of these procedures are summarized in the following subsections.

5.2.1 Non-hazardous Materials Shipment

If the samples are suspected to be non-hazardous based on previous site sample results, field screening results, or visual observations, if applicable, then samples may be shipped as non-hazardous.

When a cooler is ready for shipment to the laboratory, prepare standard air bill paperwork for shipment of the samples to the laboratory. Write the shippers tracking/airbill number on the COC form. Place two copies of the COC form inside a self-sealing bag and tape it to the inside of the cooler. Seal the cooler with waterproof tape and label it with "Fragile," "This-End-Up" (or directional arrows pointing up), or other appropriate notices. Affix a label stating the destination (laboratory address) to each cooler. Personnel should be aware of carrier weight or other policy restrictions.

5.2.2 Hazardous Materials Shipment

Shipment of Hazardous Material is not covered in this SOP; all samples handled under this SOP are anticipated to be non-hazardous or not dangerous goods. The CTO Manager, or designee, is responsible for determining if samples collected during a specific field investigation meet the definitions for dangerous goods. If a sample is collected of a material that is listed in the Dangerous Goods List, Section 4.2, of International Air Transport Authority (IATA), then that sample must be identified, packaged, marked,

labeled, and shipped according to the instructions given for that material. If the composition of the collected sample(s) is unknown, and the project leader knows or suspects that it is a regulated material (dangerous goods), the sample may not be offered for air transport. If the composition and properties of a waste sample or a highly contaminated soil, sediment, or water sample are unknown, or only partially known, the sample may not be offered for air transport.

6.0 Records

Maintain all copies of chain of custodies and air bills with the project file.

7.0 Attachments or References

International Air Transport Authority (IATA). Dangerous Goods Regulations

http://www.iata.org/whatwedo/cargo/dangerous_goods/Documents/DGR52-significant-changes.pdf

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Author	Reviewer	Revisions (Technical or Editorial)
Ben Brantley	Tina Cantwell	Rev 0 — Initial Issue
Program Manager	QA Officer	

Investigation Derived Waste Management

Procedure 3-05

1.0 Purpose and Scope

This standard operating procedure (SOP) describes activities and responsibilities of the United States (U.S.) Navy Environmental Restoration (ER) Program, Naval Facilities Engineering Command, Atlantic (NAVFAC Atlantic) with regard to management of investigation-derived waste (IDW). The purpose of this procedure is to provide guidance for the minimization, handling, labelling, temporary storage, inventory, classification, and disposal of IDW generated under the ER Program. This procedure will also apply to personal protective equipment (PPE), sampling equipment, decontamination fluids, non-IDW trash, non-indigenous IDW, and hazardous waste generated during implementation of removal or remedial actions. The information presented will be used to prepare and implement work plans (WPs) for IDW-related field activities. The results from implementation of WPs will then be used to develop and implement final IDW disposal plans.

If there are procedures whether it be from Resolution Consultants, state and/or federal that are not addressed in this SOP and are applicable to IDW then those procedures may be added as an appendix to the project specific SAP.

This procedure applies to all Navy ER projects performed in the NAVFAC Atlantic Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the Uniform Federal Policy-Quality Assurance Project Plan (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved by both the Contract Task Order (CTO) Manager and the Quality Assurance (QA) Manager or Technical Director, and documented.

This procedure was developed to serve as management-approved professional guidance for the management of IDW generated under the ER Program. It focuses on the requirements for minimizing, segregating, handling, labeling, storing, and inventorying IDW in the field. Certain drum inventory requirements related to the screening, sampling, classification, and disposal of IDW are also noted in this procedure.

2.0 Safety

The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the CTO WP and/or direction from the **Site Safety Officer (SSO)**.

All **Field Personnel** responsible for IDW management must adhere to the HASP and must wear the PPE specified in the site-specific HASP. Generally, this includes, at a minimum, steel-toed boots or steel-toed rubber boots, safety glasses, American National Standards Institute-standard hard hats, and hearing protection (if heavy equipment is in operation). If safe alternatives are not achievable, discontinue site activities immediately.

3.0 Terms and Definitions

None.

4.0 Training and Qualifications

- 4.1 The **CTO Manager** is responsible for ensuring that IDW management activities comply with this procedure. The **CTO Manager** is responsible for ensuring that all personnel involved in IDW management shall have the appropriate education, experience, and training to perform their assigned tasks.
- 4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.
- 4.3 The **Field Manager** is responsible for ensuring that all IDW is managed according to this procedure.
- 4.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.0 Equipment and Supplies

The equipment and supplies required for implementation of this SOP include the following:

- Containers for waste (e.g., [U.S. Department of Transportation] DOT approved 55-gallon open and closed top drums) and material to cover waste to protect from weather (e.g., plastic covering);
- Hazardous /non-hazardous waste drum labels (weatherproof);
- Permanent marking pens;
- Inventory forms for project file;
- Plastic garbage bags, zip lock storage bags, roll of plastic sheeting; and
- Steel-toed boots, chemical resistant gloves, coveralls, safety glasses, and any other PPE required in the HASP.

6.0 Procedure

The following procedures are used to handle the IDW.

6.1 Drum Handling

- 6.1.1 IDW shall be containerized using DOT approved drums. The drums shall be made of steel or plastic, have a 55-gallon capacity, be completely painted or opaque, and have removable lids (i.e., United Nations Code 1A2 or 1H2). Typically 55-gallon drums are used, however small drums may be used depending on the amount of waste generated. New steel drums are preferred over recycled drums.
- 6.1.2 Recycled drums should not be used for hazardous waste, PCBs or other regulated shipments. For short-term storage of liquid IDW prior to discharge, double-walled bulk steel or plastic storage tanks may be used. For this scenario, consider the scheduling and cost-effectiveness of this type of bulk storage, treatment, and discharge system versus longer-term drum storage.
- 6.1.3 For long-term IDW storage at other project locations, the DOT approved drums with removable lids are recommended. Verify the integrity of the foam or rubber sealing ring located on the underside of some drum lids prior to sealing drums containing IDW liquids.
- 6.1.4 If the ring is only partially attached to the drum lid, or if a portion of the ring is missing, select another drum lid with a sealing ring that is in sound condition.
- 6.1.5 To prepare IDW drums for labeling, wipe clean the outer wall surfaces and drum lids of all material that might prevent legible and permanent labeling. If potentially contaminated material adheres to the outer surface of a drum, wipe that material from the drum, and segregate the paper towel or rag used to remove the material with visibly soiled PPE and

disposable sampling equipment. Label all IDW drums and place them on pallets prior to storage.

6.2 Labelling

- 6.2.1 Containers used to store IDW must be properly labelled. Two general conditions exist: 1) from previous studies or on-site data, waste characteristics are known to be either hazardous or nonhazardous; or 2) waste characteristics are unknown until additional data are obtained.
- 6.2.2 For situations where the waste characteristics are known, the waste containers should be packaged and labelled in accordance with state regulations and any federal regulations that may govern the labelling of waste.
- 6.2.3 The following information shall be placed on all non-hazardous waste labels:
- Description of waste (i.e., purge water, soil cuttings);
 - Contact information (i.e., contact name and telephone number);
 - Date when the waste was first accumulated.
- 6.2.4 The following information shall be placed on all hazardous waste labels:
- Description of waste (i.e., purge water, soil cuttings);
 - Generator information (i.e., name, address, contact telephone number);
 - EPA identification number (supplied by on-site client representative);
 - Date when the waste was first accumulated.
- 6.2.5 When the final characterization of a waste is unknown, a notification label should be placed on the drum with the words "waste characterization pending analysis" and the following information included on the label:
- Description of waste (i.e., purge water, soil cuttings);
 - Contact information (i.e., contact name and telephone number);
 - Date when the waste was first accumulated.
- 6.2.6 Once the waste has been characterized, the label should be changed as appropriate for a nonhazardous or hazardous waste.
- 6.2.7 Waste labels should be constructed of a weatherproof material and filled out with a permanent marker to prevent being washed off or becoming faded by sunlight. It is recommended that waste labels be placed on the side of the container, since the top is more subject to weathering. However, when multiple containers are accumulated together, it also may be helpful to include labels on the top of the containers to facilitate organization and disposal.
- 6.2.8 Each container of waste generated shall be recorded in the field notebook used by the person responsible for labelling the waste. After the waste is disposed of, either by transportation off-site or disposal on-site in an approved disposal area, an appropriate record shall be made in the same field notebook to document proper disposition of IDW.

6.3 **Types of Site Investigation Waste**

Several types of waste are generated during site investigations that may require special handling. These include solid, liquid, and used PPE, as discussed further below.

Solid Waste

Soil cuttings from boreholes will typically be placed in containers unless site specific requirements allow for soil cuttings to be placed back into the borehole after drilling is complete. Drilling mud generated during investigation activities shall be collected in containers. Covers should be included on the containers and must be secured at all times and only open during filling activities. The containers shall be labelled in accordance with this SOP. An inventory containing the source, volume, and description of material put in the containers shall be logged on prescribed forms and kept in the project file.

Non-hazardous solid waste can be disposed on-site in the designated site landfill or in a designated evaporation pond if it is liquefied. Hazardous wastes must be disposed off-site at an approved hazardous waste landfill.

Liquid Waste

Groundwater generated during monitoring well development, purging, and sampling can be collected in truck-mounted containers and/or other transportable containers (i.e., 55-gallon drums). Lids or bungs on drums must be secured at all times and only open during filling or pumping activities. The containers shall be labelled in accordance with this SOP. Non-hazardous liquid waste can be disposed of in one of the designated lined evaporation ponds on-site. Hazardous wastes must be handled separately and disposed off-site at an approved hazardous waste facility.

Personal Protective Equipment

PPE that is generated throughout investigation activities shall be placed in plastic garbage bags. If the solid or liquid waste that was being handled is characterized as hazardous waste, then the corresponding PPE should also be disposed as hazardous waste. If not, all PPE should be disposed as non-hazardous waste in the designated on-site landfill. Trash that is generated as part of field activities may be disposed of in the landfill as long as the trash was not exposed to hazardous media.

6.4 **Waste Accumulation On-Site**

- 6.4.1 Solid, liquid, or PPE waste generated during investigation activities that are classified as nonhazardous or "characterization pending analysis" should be disposed of as soon as possible. Until disposal, such containers should be inventoried, stored as securely as possible, and inspected regularly, as a general good practice.
- 6.4.2 Solid, liquid, or PPE waste generated during investigation activities that are classified as hazardous shall not be accumulated on-site longer than 90 days. All hazardous waste containers shall be stored in a secured storage area. The following requirements for the hazardous waste storage area must be implemented:
 - Proper hazardous waste signs shall be posted as required by any state or federal statutes that may govern the labelling of waste;
 - Secondary containment to contain spills;
 - Spill containment equipment must be available;
 - Fire extinguisher;
 - Adequate aisle space for unobstructed movement of personnel.

- 6.4.3 Weekly storage area inspections shall be performed and documented to ensure compliance with these requirements. Throughout the project, an inventory shall be maintained to itemize the type and quantity of the waste generated.

6.5 Waste Disposal

- 6.5.1 Solid, liquid, and PPE waste will be characterized for disposal through the use of client knowledge, laboratory analytical data created from soil or groundwater samples gathered during the field activities, and/or composite samples from individual containers.
- 6.5.2 All waste generated during field activities will be stored, transported, and disposed of according to applicable state, federal, and local regulations. All wastes classified as hazardous will be disposed of at a licensed treatment storage and disposal facility or managed in other approved manners.
- 6.5.3 In general, waste disposal should be carefully coordinated with the facility receiving the waste. Facilities receiving waste have specific requirements that vary even for non-hazardous waste, so characterization should be conducted to support both applicable regulations and facility requirements.

6.6 Regulatory Requirements

The following federal and state regulations shall be used as resources for determining waste characteristics and requirements for waste storage, transportation, and disposal:

- Code of Federal Regulations (CFR), Title 40, Part 261;
- CFR, Title 49, Parts 172, 173, 178, and 179.

6.7 Waste Transport

A state-certified hazardous waste hauler shall transport all wastes classified as hazardous. Typically, the facility receiving any waste can coordinate a hauler to transport the waste. Shipped hazardous waste shall be disposed of in accordance with all RCRA/USEPA requirements. All waste manifests or bills of lading will be signed either by the client or the client's designee.

7.0 Quality Control and Assurance

- 7.1 Management of IDW must incorporate quality control measures to ensure conformance to these and the project requirements.

8.0 Records, Data Analysis, Calculations

- 8.1 Maintain records as required by implanting the procedures in this SOP.
- 8.2 Deviations from this procedure or the sampling and analysis plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

9.0 Attachments or References

Department of Defense, United States (DoD). 2005. [*Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*](#). Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/-fedfac/pdf/ufp_qapp_v1_0305.pdf.

Department of Energy, United States (DOE). 1994. [*The Off-Site Rule*](#). EH-231-020/0194. Office of Environmental Guidance. March.

1999. *Management of Remediation Waste under the Resource Conservation and Recovery Act (RCRA)*. Office of Environmental Policy and Assistance. 20 December.

Department of the Navy (DON). 2001. [*Department of the Navy Installation Restoration Manual. 2001 Update*](#). Draft. Alexandria, VA: Naval Facilities Engineering Command. August.

2007. [*Navy Environmental and Natural Resources Program Manual*](#). OPNAV Instruction 5090.1c . October.

Environmental Protection Agency, United States (EPA). 1991. *Management of Investigative-Derived Wastes During Site Inspections*. Office of Emergency and Remedial Response. EPA/540/G-91/009. May.

1992a. *Guidance for Performing Site Inspections under CERCLA*. [EPA/540/R-92/021](#). Office of Emergency and Remedial Response. September.

1992b. *Guide to Management of Investigative-Derived Wastes*. Quick reference fact sheet. OSWER Dir. 9345.3-03FS. Office of Solid Waste and Emergency Response. January.

1997a. *Sending Wastes Off Site? OSC and RPM Responsibilities under the Off-Site Rule*. EPA/540-F-97-006, Office of Solid Waste and Emergency Response. September.

1997b. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846*. 3rd ed., Final Update IIIA. Office of Solid Waste. Updates available: www.epa.gov/epaoswer/hazwaste/test/new-meth.htm.

1998. *Management of Remediation Waste under RCRA*. EPA/530-F-98-026. Office of Solid Waste and Emergency Response. October.

(No Date). *Compliance with the Off-Site Rule During Removal Actions*. Office of Regional Counsel (Region 3). Hendershot, Michael.

NAVFAC NW Standard Operating Procedure Number I-D-1, *Drum Sampling*.

NAVFAC NW Standard Operating Procedure Number I-F, *Equipment Decontamination*.

NAVFAC NW Standard Operating Procedure Number III-D, *Logbooks*.

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)

Land Surveying

Procedure 3-07

1.0 Purpose and Scope

- 1.1 The purpose of this document is to define the standard operating procedure (SOP) for acquiring land surveying data to facilitate the location and mapping of geologic, hydrologic, geotechnical data, and analytical sampling points and to establish topographic control over project sites.
- 1.2 This procedure is the Program-approved professional guidance for work performed by Resolution Consultants under the Comprehensive Long-Term Environmental Action Navy (CLEAN) contract (Contract Number N62470-11-D-8013).
- 1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review. If there are procedures whether it be from Resolution Consultants, state and/or federal that are not addressed in this SOP and are applicable to surface water sampling then those procedures may be added as an appendix to the project specific SAP.
- 1.4 It is fully expected that the procedures outlined in this SOP will be followed. Procedural modifications may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by the Program Quality Manager. Deviations to this SOP will be documented in the field records.
- 1.5 If there are procedures, whether it be from Resolution Consultants, state and/or federal, that are not addressed in this SOP and are applicable to land surveying then those procedures may be added as an appendix to the project specific Sampling and Analysis Plan (SAP).

2.0 Safety

- 2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to conducting fieldwork. All **field sampling personnel** must review the project-specific health and safety plan (HASP) paying particular attention to the control measures planned for the specific field tasks. Conduct preliminary area monitoring to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor and liquid phase through the use of respirators and disposable clothing.
- 2.2 In addition, observe standard health and safety practices according to the project-specific HASP. Suggested minimum protection includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves, rubberized steel-toed boots, and an American National Standards Institute-standard hard hat. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations, and shall always be available on site.
- 2.3 Daily safety briefs will be conducted at the start of each working day before any work commences. These daily briefs will be facilitated by the **Site Safety Officer (SSO)** or designee to discuss the day's events and any potential health risk areas covering every aspect of the work to be completed. Weather conditions are often part of these discussions. As detailed in the HASP, everyone on the field team has the authority to stop work if an unsafe condition is perceived until the conditions are fully remedied to the satisfaction of the SSO.
- 2.4 The health and safety considerations for the work associated with land surveying include:
 - Slip, trips and falls associated with work in the field;

- Biological hazards associated with work in the field; and,
- Potential hazards associated with contaminants of concern (COC) that may be located in the survey area,

3.0 Terms and Definitions

3.1 Boundary Survey

Boundary surveys are conducted by Certified Land Surveyors in order to delineate a legal property line for a site or section of a site.

3.2 Global Positioning System (GPS)

A system of satellites, computers, and receivers that is able to determine the latitude and longitude of a receiver on Earth by calculating the time difference for signals from different satellites to reach the receiver.

4.0 Interferences

- 4.1 Commercially available GPS units typically have a level of precision of (\pm) 3 to 5 meters. Field corrections can be made as described in Section 8.3 below.

5.0 Training and Qualifications

5.1 Qualifications and Training

- 5.1.1 The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

- 5.2.1 The **Contract Task Order (CTO) Manager** is responsible for ensuring that land surveying activities comply with this procedure. The CTO Manager is responsible for ensuring that all field sampling personnel involved in land surveying shall have the appropriate education, experience, and training to perform their assigned tasks.
- 5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.
- 5.2.3 The **Field Manager (FM)** is responsible for ensuring that all field personnel follow these procedures. In virtually all cases, subcontractors will conduct these procedures. The FM or designee is responsible for overseeing the activities of the subcontractor and ensuring that sampling points and topographic features are properly surveyed.

6.0 Equipment and Supplies

- 6.1 The following equipment list contains materials that may be needed in carrying out the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.
- Personal protective equipment (PPE) and other safety equipment, as required by the HASP;
 - Commercially available GPS unit; and,
 - Field Logbook.

7.0 Calibration or Standardization

- 7.1 An authorized manufacturer's representative shall inspect and calibrate survey instruments in accordance with the manufacturer's specifications regarding procedures and frequencies. At a minimum, instruments shall be calibrated no more than six months prior to the start of the survey work.
- 7.2 Standards for all survey work shall be in accordance with National Oceanic and Atmospheric Administration standards and, at a minimum, with accuracy standards set forth below. The horizontal accuracy for the location of all grid intersection and planimetric features shall be (\pm) 0.1 feet. The horizontal accuracy for boundary surveys shall be 1 in 10,000 feet (1:10,000). The vertical accuracy for ground surface elevations shall be (\pm) 0.1 feet. Benchmark elevation accuracy and elevation of other permanent features, including monitoring wellheads, shall be (\pm) 0.01 feet.

8.0 Procedure

8.1 Theodolite/Electronic Distance Measurement (EDM)

Follow the procedures listed below during theodolite/EDM land surveying conducted under the NAVFAC CLEAN Program:

- A land surveyor registered in the state or territory in which the work is being performed shall directly supervise all surveying work.
- Reference surveys to the local established coordinate systems and base all elevations and benchmarks established on U.S. Geological Survey datum, 1929 general adjustment.
- Reference surveyed points to Mean Sea Level (Lower Low Water Level).
- Jointly determine appropriate horizontal and vertical control points prior to the start of survey activities. If discrepancies in the survey (e.g., anomalous water level elevations) are observed, the surveyor may be required to verify the survey by comparison to a known survey mark. If necessary, a verification survey may be conducted by a qualified third party.
- All field notes, sketches, and drawings shall clearly identify the horizontal and vertical control points by number designation, description, coordinates, and elevations. Map all surveyed locations using a base map or other site mapping, as specified by the project Work Plan or SAP.
- Begin and end all surveys at the designated horizontal and vertical control points to determine the degree of accuracy of the surveys.
- Iron pins used to mark control points shall be made of reinforcement steel or an equivalent material and shall be 18 inches long with a minimum diameter of 5/8 inch. Drive pins to a depth of 18 inches into the soil.
- Stakes used to mark survey lines and points shall be made from 3-foot lengths of 2-inch by 2-inch lumber and pointed at one end. Clearly mark them with brightly colored weatherproof flagging and paint.
- Clearly mark the point on a monitoring well casing or well riser that is surveyed by filing grooves into the casing/riser on either side of the surveyed point, or by marking the riser with a permanent ink marker.

8.2 Global Positioning System (GPS) to Conduct Land Survey

Follow the procedures listed below during land surveying using GPS:

- A land surveyor registered in the state or territory in which the work is being performed shall directly supervise all surveying work.
- Reference surveys to the local established coordinate systems and base all elevations and benchmarks established on U.S. Geological Survey datum, 1929 general adjustment.

- All field notes, sketches, and drawings shall clearly identify the horizontal and vertical control points by number designation, description, coordinates, and elevations. Map all surveyed locations using a base map or other site mapping, as specified in the project Work Plan or SAP.
- Begin and end all surveys at the designated horizontal and vertical control points (as applicable) to determine the degree of accuracy of the surveys.
- Iron pins used to mark control points shall be made of reinforcement steel or an equivalent material and shall be 18 inches long with a minimum diameter of 5/8 inch. Drive pins to a depth of 18 inches into the soil.
- Stakes used to mark survey lines and points shall be made from 3-foot lengths of 2-inch by 2-inch lumber and pointed at one end. Clearly mark them with brightly colored weatherproof flagging and paint.
- Clearly mark the point on a monitoring well casing that is surveyed by filing grooves into the casing on either side of the surveyed point.

8.3 **Global Positioning System (GPS) to Position Sample Locations or Locate Site Features**

Experienced field personnel may use a GPS system unit to position sample locations (e.g. grid positioned samples, soil boring locations) at a site. The decision to use field personnel or a licensed land surveyor will depend on the objectives of the survey (e.g. vertical elevation is not required) and the levels of precision required. Typically when a level of precision greater than (\pm) 3 to 5 meters is required, a licensed surveyor will be required. When a level of precision of (\pm) 3 to 5 meters is sufficient to meet project requirements (i.e. when laying sampling grids, identifying significant site features, or locating features identified in GIS figures) experienced field personnel may use commercially available, consumer-grade GPS units. Follow the procedures listed below to locate samples or site features using GPS:

- A commercially available GPS unit with Wide Angle Averaging System (WAAS), topographic map display, and waypoint storage capabilities should be used.
- If waypoints are to be imported into a GIS database, the same grid projection system should be used.
- If a permanent reference point near the site is available, it is recommended that a waypoint at this location be taken every day waypoints are stored.
- When laying out a sampling grid from a GIS map, upload the coordinates from GIS to the GPS unit, including coordinates for an easily identified, permanent, nearby feature (i.e. building corner, roadway intersection, or USGS benchmark).
- If during the initial site walk, the permanent feature identified does not overlay within (\pm) 5 meters as identified in the GPS unit, field corrections of the waypoints should be made.
- Field corrections can be made by adding/subtracting the difference in x,y coordinates between the field measurement of the permanent site feature and the anticipated x,y coordinates. This correction should then be applied to the x,y coordinates for each sampling location to be marked. Corrected x,y coordinates can then be uploaded into the GPS unit.
- Sampling points and site features can then be located in the field using the GPS units "Go To" function. When the distance to the sampling point or feature remains close to zero, the location can be marked.
- If no field corrections to the sampling location need to be made, or if sampling locations are to be surveyed by a licensed surveyor at a later date, no additional waypoints need to be taken. If significant changes to the sampling location are made, GPS coordinates at the corrected location shall be stored and labeled.

- It is recommended that GPS coordinates be uploaded to a storage device such as PC at the end of each day.
- Field logs shall indicate manufacturer and model number for GPS unit used, map datum and projection used, and any field corrections made. If the GPS unit cannot lock onto a WAAS system at the site, this should also be noted.

9.0 Quality Control and Assurance

None.

10.0 Data and Records Management

The surveyor shall record field notes daily using generally accepted practices. The data shall be neat, legible, in indelible ink, and easily reproducible. Copies of the surveyor's field notes and calculation forms generated during the work shall be obtained and placed in the project files.

Surveyor's field notes shall, at a minimum, clearly indicate:

- The date of the survey;
- General weather conditions;
- The name of the surveying firm;
- The names and job titles of personnel performing the survey work;
- Equipment used, including serial numbers; and,
- Field book designations, including page numbers.

A land surveyor registered in the state or territory in which the work was done shall sign, seal, and certify the drawings and calculations submitted by the surveyor.

Dated records of land surveying equipment calibration shall be provided by the surveyor and placed in the project files. Equipment serial numbers shall be provided in the calibration records.

11.0 Attachments or References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

<i>Author</i>	<i>Reviewer</i>	<i>Revisions (Technical or Editorial)</i>
Robert Shoemaker Senior Scientist	Naomi Ouellette, Project Manager	Rev 0 – Initial Issue

Monitoring Well Installation

Procedure 3-12

1.0 Purpose and Scope

- 1.1 This standard operating procedure (SOP) describes the methods to be used during the installation of groundwater monitoring wells. It describes the components of monitoring well design and installation and sets forth the rationale for use of various well installation techniques in specific situations.
- 1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

- 2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the Contract Task Order (CTO) Work Plan (WP) and/or direction from the **Site Safety Officer (SSO)**.
- 2.2 Before well installation commences, appropriate entities (e.g. DigSafe, local public works departments, company facilities) must be contacted to assure the anticipated well locations are marked for utilities, including electrical, telecommunications, water, sewer, and gas.
- 2.3 Physical Hazards Associated with Well Installation
- Stay clear of all moving equipment and avoid wearing loose fitting clothing.
 - When using an approved retractable-blade knife, always cut away from one self and make sure there are no other people in the cutting path or the retractable-blade knife.
 - To avoid slip/trip/fall conditions during drilling activities, keep the area clear of excess soil cuttings and groundwater. Use textured boots/boot cover bottoms in muddy areas.
 - To avoid heat/cold stress as a result of exposure to extreme temperatures and personal protective equipment (PPE), drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and, in cases of extreme cold, wear fitted insulating clothing.
 - To avoid hazards associated with subsurface utilities, ensure all sampling locations have been properly surveyed as described in SOP 3-01, Utility Clearance.
 - Be aware of restricted mobility caused by PPE.

3.0 Terms and Definitions

- 3.1 **Annulus:** The annulus is the down-hole space between the borehole wall and the well casing and screen.
- 3.2 **Bridge:** A bridge is an obstruction in the drill hole or annulus. A bridge is usually formed by caving of the wall of the well bore, by the intrusion of a large boulder, or by the placement of filter pack materials during well completion. Bridging can also occur in the formation during well development.
- 3.3 **Filter Pack:** Filter pack is sand or gravel that is smooth, uniform, clean, well-rounded, and siliceous. It is placed in the annulus of the well between the borehole wall and the well screen to prevent formation materials from entering the well and to stabilize the adjacent formation.
- 3.4 **Grout:** Grout is a fluid mixture of cement and water that can be forced through a tremie pipe and emplaced in the annular space between the borehole and casing to form an impermeable seal. Various additives, such as sand, bentonite, and polymers, may be included in the mixture to meet certain requirements.
- 3.5 **Heaving (Running) Sands:** Loose sands in a confined water-bearing zone or aquifer which tend to rise up into the drill stem when the confining unit is breached by the drill bit. Heaving sands occur when the water in the aquifer has a pressure head great enough to cause upward flow into the drill stem with enough velocity to overcome the weight of the sand.
- 3.6 **Sieve Analysis:** Sieve analysis is the evaluation of the particle-size distribution of a soil, sediment, or rock by measuring the percentage of the particles that will pass through standard sieves of various sizes.

4.0 Interferences

- 4.1 Heaving sands may be problematic in unconsolidated sands encountered below the water table.
- 4.2 Rotary drilling methods requiring bentonite-based drilling fluids should be used with caution to drill boreholes that will be used for monitoring well installation. The bentonite mud builds up on the borehole walls as a filter cake and permeates the adjacent formation, potentially reducing the permeability of the material adjacent to the well screen.
- 4.3 If water or other drilling fluids have been introduced into the boring during drilling or well installation, samples of these fluids should be obtained and analyzed for chemical constituents that may be of interest at the site. In addition, an attempt should be made to recover the quantity of fluid or water that was introduced, either by flushing the borehole prior to well installation and/or by overpumping the well during development.
- 4.4 Track-mounted drill rigs are suitable for travelling on many types of landscapes that truck-mounted units cannot access, but may have limitations on extremely uneven or soft terrain.
- 4.5 Care should be taken to prevent cross-contamination between well locations. All drilling equipment coming in contact with potentially contaminated soil and/or groundwater will be decontaminated by the drilling subcontractor prior to initial drilling activities and between drilling locations in accordance with SOP 3-06, Equipment Decontamination.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

- 5.2.1 **Contract Task Order (CTO) Managers** are responsible for issuing sampling and analysis plans (SAPs) that reflect the procedures and specifications presented in this procedure. Individual municipalities, county agencies, and possibly state regulatory agencies enforce regulations that may include well construction and installation requirements. The **CTO Manager** shall be familiar with current local and state regulations, and ensure that these regulations are followed. The **CTO Manager** is responsible for ensuring that all personnel involved in monitoring well installation shall have the appropriate education, experience, and training to perform their assigned tasks.
- 5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.
- 5.2.3 The **Field Manager** is responsible for direct supervision of the installation of monitoring wells and ensuring that procedures and specifications are implemented in the field in accordance with the approved SAP and well installation permits. The qualifications for the **Field Manager** must be in accordance with local jurisdictions with authority over the operations conducted.
- 5.2.4 All field personnel are responsible for the implementation of this procedure.
- 5.2.5 The on-site hydrogeologist/engineer is expected to obtain a description of the lithologic samples obtained during the excavation and construction of a monitoring well. These data are often required to provide guidance regarding the installation of specific components of the monitoring well. Guidance for lithologic sample collection and sample description is contained within SOP 3-16, Soil and Rock Classification.

6.0 Equipment and Supplies

6.1 Materials provided by the drilling contractor may include:

- Drill rig, drill rods, hollow stem augers, etc.
- Decontamination equipment (e.g., steam cleaner, high-pressure washer, brushes, etc.)
- Decontamination pad materials
- Well screen/riser pipe with flush-threaded couplings including riser and bottom caps
- Clean, filter sand
- Bentonite chips or pellets
- Cement grout and tremie pipe
- Portland cement for well pad completion
- Steel protective riser covers and locking caps
- Weighted calibrated tape
- Split-spoon samplers
- 55-gallon drums or containers for drill cuttings, decontamination fluids, etc.

6.2 In addition to those materials provided by the drilling contractor, equipment and materials required by the project geologist/engineer may include, but is not limited to, the following:

- Photoionization Detector (PID)
- Spill kit, including at a minimum sorbent pads and shovel (if not provided by subcontractor)

- Plastic sheeting
- Teaspoon or spatula
- Resealable plastic bags
- Boring Log Records
- Decontamination materials (per SOP No. 3-06 - Equipment Decontamination)
- Weighted measuring tape for depth measurement
- Soil logging materials (e.g. USCS classification field card, millimeter rule, hand lens, etc.)
- Survey lathes or pin flags
- Digital camera
- PPE as required by the HASP
- Planning documents including the site-specific HASP and SAP
- Large indelible ink or paint pen
- Field logbook/field forms/site maps (water proof)

7.0 Procedure

7.1 General Procedures

- Specific drilling, sampling, and installation equipment and methodology will be dictated by the type of well to be installed (e.g., single case (Type II), double case (Type III), bedrock, etc.), geologic characteristics of the site, the type of contaminants being monitored, and local and state regulations.
- For access to locations when travelling over difficult terrain, an appropriate line should be chosen before mobilizing the drill rig or other support vehicles. If clearing of trees or ground cover is required, perform these activities in advance to avoid down time. Avoid wet or soft areas where possible or use ground mats and/or timbers to aid in supporting the rig as it travels. If drilling on soft material, place geomatting and ground mats under the rig tracks or stabilizers prior to drilling.
- A utility locate must be conducted to identify all underground utilities at the site prior to drilling (refer to SOP 3-01, Utility Clearance). Proper clearance procedures for aboveground/overhead utilities must also be followed as specified in the HASP.
- Although new well materials (well screen and riser pipe) generally arrive at the site boxed and sealed within plastic bags, it is sometimes necessary to decontaminate the materials prior to their use. Well materials should be inspected by the project geologist/engineer upon delivery to check for cleanliness. If the well materials appear dirty, or if local or regional regulatory guidance requires decontamination, then well material decontamination should be performed by the drilling subcontractor in accordance with SOP 3-06, Equipment Decontamination.
- The diameter of the borehole must be a minimum of 2 inches greater than the outside diameter of the well screen or riser pipe used to construct the well. This is necessary so that sufficient annular space is available to install filter packs, bentonite seals, and grout seals, and allow the passage of tremie pipe where grouting at depth is required. Bedrock wells may require reaming after coring in order to provide a large enough borehole diameter for well installation.
- When soil sampling is required (refer to the SAP), soil samples will be collected for visual logging by advancing split-spoon samplers through the augers. The soil will be visually logged by a field geologist and include lithologic characteristics (i.e., soil type, color, density, moisture content, etc.) using the the

methods described in SOP 3-16, Soil and Rock Classification. This information will be recorded on a boring/well log form, along with well construction details.

7.2 Drilling Techniques

Drilling of monitoring well boreholes may be accomplished by a variety of methods as described below. Preferred methods include those that temporarily case the borehole during drilling (i.e., hollow stem auger and sonic methods) using an override system. Other methods can be used where specific subsurface conditions or well design criteria dictate.

- Hollow stem auger (HSA) – Borings are advanced by rotating steel hollow stem augers with an attached cutting head. Soil cuttings are displaced by the cutting head and transported to the surface via continuous spiral flights attached to each auger stem. This method is widely used for unconsolidated soils that have a tendency to collapse within the boring. A bottom plug can be placed in the bottom auger to prevent soils from entering and clogging the auger, especially in the case of heaving sands. However, a bottom plug cannot be used when soil samples are to be collected through the augers. Soil plugs that accumulate in the bottom of the auger must be removed or knocked out prior to sampling or well installation.
- Solid stem auger – This type of drilling method is similar to HSA drilling using a solid stem or sealed hollow stem auger flights to advance the boring. Solid stem, continuous flight auger use is limited to semi-consolidated sediments or to cohesive or semi-cohesive unconsolidated sediments that don't have a tendency to collapse when disturbed.
- Sonic methods – Sonic drilling consists of advancing concentric hollow drill casings (inner and outer) using rotation in conjunction with axial vibration of the drill casing. Once the casings are advanced to the appropriate depth, the inner string is removed with a core of drill cuttings while the outer casing remains in place to keep the borehole open. Cuttings are removed from the inner casing relatively intact for logging or sampling purposes. This drilling method is used for a variety of soil types, from heaving sands to consolidated or indurated formations. Smearing of the formation along the borehole walls is minimal since moderate vibration and rotation techniques are used to advance the casings. Since the total borehole diameter in sonic drilling is only incrementally larger than the inner casing diameter, care should be taken during installation of the monitoring well to ensure the well is centered and adequate space is available for annular materials.
- Rotary methods (water or mud) – Rotary drilling methods consist of drill rods coupled to a drill bit that rotates and cuts through the soils to advance the borehole. Water or drilling fluid ("mud") is forced through the hollow drill rods and drill bit as the rods are rotated. The soil cuttings are forced up the borehole with the drilling fluids to the surface and the fluids recirculated. The drilling fluid provides a hydrostatic pressure that reduces or prevents the borehole from collapsing. Clean, potable water must be used for water-rotary drilling to prevent introducing trace contaminants. A sample of the potable water should be collected during the course of well installation for analysis of the same parameters defined for the groundwater samples. If mud-rotary is used to advance boreholes, potable water and bentonite drilling mud should only be used. No chemical additives shall be mixed in the drilling fluid to alter viscosity or lubricating properties. Adequate well development is essential for removal of drilling mud and fluids from the formation materials and ensure collection of representative groundwater samples.
- Rotary methods (Air) – Air rotary methods are similar to water rotary but use high air velocities in place of drilling fluids to rotate the drill bit and carry the soil cuttings up the borehole to the surface. Care must be taken to ensure that contaminants are not introduced into the air stream from compressor oils, etc. Most compressor systems are compatible with a coalescing filter system. Cuttings exiting the borehole under pressure must be controlled, especially when drilling in a zone of potential contamination. This can be accomplished by using an air diverter with hose or pipe to carry the cuttings to a waste container. Letting the cuttings blow uncontrolled from the borehole is not acceptable.

7.3 Well Construction and Installation

- If rotary drilling techniques are used, the borehole should be flushed or blown free of material prior to well installation. If hollow stem augers are used, the soil or bottom plug should be removed and the augers raised approximately six inches above the bottom of the borehole, while slowly rotating the augers to remove cuttings from the bottom of the boring. The depth of the borehole should be confirmed with a weighted, calibrated tape.
- The riser pipe and screen should be connected with flush-threaded joints and assembled wearing clean, disposable gloves. No solvent or anti-seize compound should be used on the connections. The full length of the slotted portion of the well screen and unslotted riser pipe should be measured and these measurements recorded on a well construction form (Attachment 1).
- If placed in an open borehole, the assembled well should be carefully lowered and centered in the borehole so that the well is true, straight, and vertical throughout. Centering can also be accomplished with the use of centralizers, if necessary. However, centralizers should be placed so that they do not inhibit the installation of filter sand, bentonite seal, and annular grout. Wells less than 50 deep generally do not require centralizers.
- If hollow stem augers are used, the well should be lowered through the augers and each auger flight removed incrementally as the filter sand, bentonite seal, and grout are tremmied or poured into the annular space of the well. The well should be temporarily capped before filter sand and other annular materials are installed.
- Clean, silica sand should be placed around the well screen to at least 1 foot above the top of the screen. The filter sand should be appropriately graded and compatible with the selected screen size and surrounding formation materials. In general, the filter pack should not extend more than 3 feet above the top of the screen to limit the thickness of the monitoring zone. As the filter pack is placed, a weighted tape should be lowered in the annular space to verify the depth to the top of the layer. This measurement will be recorded on the well construction form (Attachment 1). If necessary, to eliminate possible bridging or creation of voids, placement of the sand pack may require the use of a tremie pipe. Tremie pipe sandpack installations are generally suggested for deeper wells and for wells which are screened some distance beneath the water table.
- A minimum 2-foot thick layer of bentonite pellets or slurry seal will be installed immediately above the filter sand to prevent vertical flow within the boring from affecting the screened interval. Bentonite chips/pellets must be hydrated if placed above the water table prior to grouting. If bridging is of concern as in the case of deep wells, powdered bentonite may be mixed with water into a very thick slurry and a tremie pipe used to place the seal to the desired depth. Placement of the bentonite seal in the borehole will be recorded on the well construction form (Attachment 1).
- The remaining annular space around the well will be grouted from the top of the bentonite seal to the surface with a grout composed of neat cement, a bentonite cement mixture, or high solids sodium bentonite grout.
- Each well riser will be secured with an expandable, locking cap (vented if possible). Optionally, a hole can be drilled in the upper portion of the riser to allow venting of the well.
- The well will be completed within a concrete well pad consisting of a Portland cement/sand mixture. Well pads are generally 3 feet by 3 feet square but may be larger or smaller depending on site conditions and state-specific well construction standards. Round concrete well pads are also acceptable. A minimum of 1 inch of the finished pad should be below grade to prevent washing and undermining by soil erosion.
- If completed as a flush-mount well, the well riser will be cut off approximately 4 to 6 inches below ground surface and an expandable, locking cap placed on the well riser. The area around the riser is dug out and a steel well vault or manhole cover placed over the riser and set almost flush to the ground.

to protect the well. The manhole cover should be water-tight and secured with bolts to prevent casual access. The well pad will then be constructed around the well vault and slightly mounded at the center and sloping away to prevent surface water from accumulating in the well vault.

- If completed as a stick-up well, the well riser is cut approximately 2.5 to 3 feet above the ground surface and an expandable, locking cap placed on the well riser. A steel guard pipe with hinged, locking cap is placed over the well riser as a protective casing. The bottom of the guard pipe will be set approximately 2 feet below ground surface and sealed by pouring concrete from the top of the annular grout around the pipe to grade. The concrete well pad should be completed at the same time. Weep holes will be drilled in the base of the guard pipe to facilitate draining of rainwater or purge water from inside the guard pipe.
- Bumper posts or bollards may be necessary for additional well protection, especially in high traffic areas. The bumper posts should be placed around the well pad in a configuration that provides maximum protection to the well and extend a minimum of 3 feet above the ground.

7.4 Double Cased Wells

Under certain site conditions, the use of a double-cased or telescoping (Type III) well may be necessary. Installation of double-cased wells may be required to prevent the interconnection of two separate aquifers, seal off a perched aquifer without creating a vertical hydraulic conduit, prevent cross-contamination during construction of wells in deeper aquifers hydro-stratigraphically below impacted aquifers, or case off highly impacted soils present above the aquifer to prevent potential “dragging down” of contaminants.

Similar to conventional wells, construction of double-cased wells can be accomplished using a variety of drilling methods. Well construction is initiated by “keying” a large diameter, outer casing into a stratigraphic zone of low permeability (clay layer or bedrock). The size of the outer casing should be a minimum of 2 inches greater than the outside diameter of the inner casing to allow installation of annular seal materials during well completion. A pilot borehole should be drilled through the overburden soil and/or contaminated zone into a clay confining layer or bedrock. The borehole for the outer casing should be of sufficient size to contain the outer casing with a minimum of 2 inches around the outside diameter to allow sufficient annular space for tremie or pressure grouting. The boring should extend a minimum of 2 feet into a clay layer and a minimum of 1 foot into bedrock, if possible, to ensure an adequate seal. The boring should never breach a confining layer or keyed zone under any circumstances.

Once the boring is completed, the outer casing can be set in the borehole and sealed with grout. The outer casing can be set two ways, with or without a bottom cap. If no bottom cap is applied, the casing is usually driven approximately 6 inches into the clay confining unit. A grout plug is generally placed in the bottom of the casing and once set, standing water in the casing is evacuated prior to drilling below the casing. As an alternative, a cap can be placed on the bottom of the casing and if set below the water table, the casing can be filled with clean, potable water to hold down the casing in the boring. Grouting should be conducted using tremie-grouting or pressure-grouting methods by pumping grout into the annular space between the outer casing and the borehole wall from the bottom of the casing to the ground surface. Grout around the casing should be allowed to cure at least 24 hours before attempting to drill through the bottom.

Once the grout is cured, a smaller diameter drill pipe/bit is used to bore through the grout plug or bottom cap to the desired well depth. The well is then constructed as described in Section 7.3 above.

7.5 Post Installation Procedures

- Wells should be permanently labelled or marked for identification. Well tags can be used to record the site name, well number, total depth, installation date, etc. At a minimum, the well number will be written in indelible marker or paint on both the outside of the protective casing and inside beneath the casing lid, as well as on the riser pipe.

- A measuring point will be marked on the top of the riser pipe for taking water level measurements. The measuring point can be notched using a knife or saw or can be marked with a waterproof marker or paint. The measuring point will also be the point which will be surveyed for vertical elevation data.
- Upon completion, the following measurements will be taken by the field geologist/engineer and recorded on the well construction diagram.
 - Depth to static water level
 - Depth of non-aqueous phase liquid (NAPL), if present
 - Total depth of well measured from top of casing (TOC)
 - Height of well casing above ground surface
 - Height of protective casing above ground surface
- All monitoring wells will be surveyed for horizontal and vertical control by a licensed surveyor.
- Investigation-derived waste (IDW) including drill cuttings, spent materials (e.g., PPE), and decontamination water should be properly managed in accordance with SOP 3-05, IDW Management.

8.0 Quality Control and Assurance

- 8.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the SAP. Certain quality control (QC) measures should be taken to ensure proper well installation and construction in accordance with this SOP, project specific SAP, and applicable well standards.
- 8.2 The borehole will be checked for total open depth, and extended by further drilling or shortened by backfilling, as required before installation of the well materials.
- 8.3 Water level and NAPL presence will be checked during well installation to ensure that the positions of well screen, filter sand, and seals relative to water level conform to project requirements
- 8.4 The depth to top of each layer of annular materials (i.e., filter sand, bentonite, grout) will be verified and adjusted as necessary for proper placement.

9.0 Records, Data Analysis, Calculations

All field information will be recorded in the field logbook and/or standardized field forms by field personnel. Field data recorded will include drilling contractor information, drilling methods, well material and construction information provided on the boring logs and well construction forms, observations or problems encountered during drilling, fluid level data, and any deviations from the procedures in this SOP and other project plans. Well Construction Forms (Attachment 1) will provide visual and descriptive information the monitoring well and are often the most critical form of documentation generated during the installation of a monitoring well. The field logbook is kept as a general log of activities and should not be used in place of the boring log.

10.0 Attachments or References


- 10.1 Attachment 1 – Monitoring Well Construction Form

- 10.2 Environmental Protection Agency, United States (EPA). 1987. *A Compendium of Superfund Field Operations Methods*. Office of Solid Waste and Emergency Response. EPA/540/P-87/001.
- 10.3 EPA. 1990. *Handbook of Suggested Practices for the Design and Installation of Groundwater Monitoring Wells*. EPA/600/4-89/034. Office of Research and Development, Washington. March.
- 10.4 EPA. 1992. *RCRA Groundwater Monitoring Draft Technical Guidance*. EPA/530/R-93/001. Office of Solid Waste. November.
- 10.5 EPA, 2008. SESD Operating Procedure SESDGUID-101-R0: *Design and Installation of Monitoring Wells*. USEPA, Science and Ecosystem Support Division (SESD), Athens, Georgia. Effective Date February 18, 2008.
- 10.6 U.S. Army Corps of Engineers. 2008. Manual No. EM 385-1-1. *Safety and Health Requirements*. 15 November 2008. http://140.194.76.129/publications/eng-manuals/em385-1-1/2008_English/toc.html.
- 10.7 SOP 3-01, *Utility Clearance*.
- 10.8 SOP 3-05, *IDW Management*
- 10.9 SOP 3-06, *Equipment Decontamination*.
- 10.10 SOP 3-16, *Soil and Rock Classification*.

<i>Author</i>	<i>Reviewer</i>	<i>Revisions (Technical or Editorial)</i>
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)

Attachment 1

Monitoring Well Construction Form

	Client: _____		WELL ID:	
	Project Number: _____			
	Site Location: _____		Date installed: _____	
	Well Location: _____ Coords: _____		Inspector: _____	
	Method: _____		Contractor: _____	

MONITORING WELL CONSTRUCTION DETAIL				
			Depth from G.S. (feet)	Elevation(feet)
				Datum _____
Measuring Point for Surveying & Water Levels	Top of Steel Guard Pipe		_____	_____
	Top of Riser Pipe		_____	_____
	Ground Surface (G.S.)		0.0	_____
Cement, Bentonite, Bentonite Slurry Grout, or Native Materials	Riser Pipe:			
	Length	_____		
	Inside Diameter (ID)	_____		
	Type of Material	_____		
Bottom of Steel Guard Pipe				
% Cement				
% Bentonite				
% Native Materials				
	Top of Bentonite		_____	_____
	Bentonite Seal Thickness	_____	_____	_____
	Top of Sand		_____	_____
	Top of Screen		_____	_____
	▼ Stabilized Water Level		_____	_____
	Screen:			
	Length	_____		
	Inside Diameter (ID)	_____		
	Slot Size	_____		
	Type of Material	_____		
	Type/Size of sand	_____		
	Sand Pack Thickness	_____		
	Bottom of Screen		_____	_____
	Bottom of Tail Pipe:		_____	_____
	Length	_____		
	Bottom of Borehole		_____	_____
Borehole Diameter		_____		
Approved:		_____		
Describe Measuring Point:		Signature _____	Date _____	

Monitoring Well Development

Procedure 3-13

1.0 Purpose and Scope

- 1.1 This standard operating procedure (SOP) describes the procedures used for developing newly installed monitoring wells and/or redeveloping existing wells.
- 1.2 The purpose of well development is to remove interferences from a well to provide better connection between the well and the formation, to improve pumping performance of the well, and to be able to collect more representative information from the well (e.g., samples, test results, etc.). Proper well development will:
- Remove drilling residuals (e.g., water, mud) from the borehole and surrounding formations;
 - Improve or restore hydraulic conductivity of the surrounding formations which may have been disturbed during the drilling process;
 - Remove residual fines from the well screen and sand pack (filter pack) materials, thus reducing turbidity of groundwater and permitting the collection of more representative groundwater samples.
- 1.3 There may be circumstances where well development is not desirable, for example, in the presence of non-aqueous phase liquids (NAPL) or other significant contamination if development could worsen the contaminant impact. If NAPL begins to intrude during development, the development process will be halted. This situation will be considered a cause for sample modification requiring approval by the CTO Manager and other stakeholders, as applicable.
- 1.4 The applicable well development procedures for a particular site may be subject to State or local regulatory requirements. In all cases, the project team should consult their local regulatory requirements and document the selected well development procedure in the project-specific Sampling and Analysis Plan (SAP). For project-specific information refer to the SAP, which takes precedence over these procedures.
- 1.5 This procedure is the Program-approved professional guidance for work performed by Resolution Consultants under the Comprehensive Long-Term Environmental Action Navy (CLEAN) contract (Contract Number N62470-11-D-8013).
- 1.6 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

- 2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the Contract Task Order (CTO) SAP and/or direction from the Site Safety Officer (SSO).
- 2.2 Monitoring well development may involve chemical hazards associated with potential contaminants in the soil or aquifer being characterized and may involve physical hazards associated with use of well development equipment.

3.0 Terms and Definitions

None.

4.0 Interferences

- 4.1 Equipment/materials used for development may react with the groundwater during development. Appropriate development equipment has been selected for the anticipated condition of the groundwater.
- 4.2 Appropriate development methods such as using a surge-block to flush suspended fines in the groundwater in and out of the well screen can improve the yield of wells and improve their potential to be developed successfully. However, the effectiveness of development can be significantly reduced in wells that do not yield sufficient water to allow this flushing to take place.
- 4.3 For formations with a significant content of fine-grained materials (silts and clays), or wells with improperly sized screens, it may not be possible to reduce turbidity to commonly acceptable levels. Possible solutions may include collecting a sample even if excessively turbid, or installing a replacement well.
- 4.4 Development itself disturbs the surrounding formation and disrupts equilibrium conditions within the well. Groundwater samples will not be collected until a minimum of 24 hours after a well is developed to allow conditions to stabilize. For sites with fine-grained formations (silts and clays) and highly sorptive contamination, a longer time period between development and sampling should be considered.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

- 5.2.1 The **CTO Manager** is responsible for ensuring that well development activities comply with this procedure. The **CTO Manager** is responsible for ensuring that all personnel involved in well development shall have the appropriate education, experience, and training to perform their assigned tasks.
- 5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.
- 5.2.3 The **Field Manager** is responsible for ensuring that all well development activities are conducted according to either this procedure or the applicable procedure presented in the project-specific SAP.
- 5.2.4 **Field sampling personnel** are responsible for the implementation of this procedure.
- 5.2.5 The field sampler and/or task manager is responsible for directly supervising the well development procedures to ensure that they are conducted according to this procedure and for recording all pertinent data collected during sampling.

6.0 Equipment and Supplies

- 6.1 This equipment list was developed to aid in field organization and should be used in planning and preparation. Depending on the site-specific requirements and the development method selected, additional or alternative material and equipment may be necessary. In addition, for sites where groundwater is expected to be contaminated, the materials to be placed down the well and in contact with groundwater should be evaluated so that they are compatible with the chemical conditions expected in the well.
- 6.2 Equipment and materials used for well development may include, but is not limited to:

Well development equipment

- Surge block

- Disposable Teflon bailers, appropriate to the diameter of the well(s): 1-inch to 1.5-inch for 2-inch inside diameter (ID) monitoring wells.
- Watterra® footvalve
- Electric submersible pump
- 12-volt power source for electric pump
- High density polyethylene (HDPE) tubing appropriately sized for Watterra® footvalve and/or electric submersible pump
- Drums or containers for storage of purge water
- Nephelometer to measure turbidity
- Multi-parameter water quality meter(s) to measure temperature, pH, conductivity, dissolved oxygen (DO), oxidation reduction potential (ORP)
- Instrument calibration solutions
- Water level meter
- Oil/water interface probe

General equipment

- Project-specific plans including the site-specific HASP and SAP
- Field notebook/field forms/site maps
- Indelible markers/pens
- 5-gallon buckets

Equipment decontamination supplies (refer to SOP 3-06, Equipment Decontamination)

- Health and safety supplies, including personal protective equipment (PPE)
- Appropriate hand tools
- Keys or combinations to access monitoring wells
- Distilled/deionized water supply
- Disposable bailer string (polypropylene)
- Plastic trash bags

7.0 Procedure

Development generally consists of removing water and entrained sediment from the well until the water is clear (to the extent feasible) and the turbidity is reduced, which indicates the well is in good hydraulic connection with the surrounding formation. In addition to simply removing water, development can be improved when flushing through the well screen and gravel pack takes place in both directions, that is, both into the well and into the formation. This action breaks down sediment bridges that can occur in the formation or sand pack, which reduce the connection between the well and the formation

7.1 General Preparation

- All down-well equipment should be decontaminated prior to use and between well locations in accordance with SOP 3-06, Equipment Decontamination
- Although equipment is decontaminated between well locations, if wells are known or suspected to be contaminated based on observations during well installation, it is recommended that well development be conducted in order from the least contaminated to the most contaminated well to minimize the chances of cross-contamination.
- Management of investigation-derived waste (IDW), including development purge water and miscellaneous expendable materials generated during the development process, will be conducted in accordance with SOP 3-05, IDW Management.

- Prior to accessing the well, the wellhead should be cleared of debris and/or standing water. Nothing from the ground surface should be allowed to enter the well.
- The depth to water and total well depth should be measured with a water level meter and recorded in the field logbook or on a Well Development Record (Attachment 1). This information will be used to calculate the volume of standing water (i.e., the well volume) within the well, and plan the specific details of the well development. If wells are suspected to contain NAPL, an oil/water interface probe should be used to measure liquid levels and depth to bottom of the well.
- Permanent monitoring wells will be developed no sooner than 24 hours after well installation is completed in order to allow well completion materials to set properly.

7.2 Monitoring Well Development Procedures

Generally, development will begin by gently surging the well with a surge block or bailer as described in Sections 7.2.1 and 7.2.2, respectively. Surging can become more vigorous as development progresses but initially the well must be gently surged to allow material blocking the screen to become suspended without damaging the well. Next, a bailer can be used to remove the sediment settled at the base of the well. A bailer, Watterra[®] pump, or electric submersible pump will then be used to purge the well, per Sections 7.2.2, 7.2.3, or 7.2.4, respectively. The well will be purged until the removed water becomes less turbid or per the requirements of the project-specific SAP, or State or local requirements. At this point the well will be surged again with a surge block or bailer. The well can be surged more vigorously at this point. After surging, the well will be purged again until the turbidity once again decreases. The surge/purge cycle should be completed at least three times during the development process. After the last surge, the well will be purged until the development completion criteria outlined in 7.3.2 or per the project-specific SAP are met.

7.2.1 Surge Block

The default method of well development is the use of a surge block in conjunction with pumping or bailing to remove sediment-laden water.

- The construction of the surge block must be appropriate for the diameter of the well. The surge block must be mounted on rods or other stiff materials to extend it to the appropriate depths and to allow for the surge block to be moved up and down in the well.
- Insert the surge block into the well and lower it slowly to the screened or open interval below the static water level. Start the surge action by slowly and gently moving the surge block up and down in the well. A slow initial surging, using plunger strokes of approximately 1 meter or 3 feet, will allow material which is blocking the screen to separate and become suspended.
- After 5 to 10 plunger strokes, remove water from the well using a separate bailer (Section 7.2.2) or pumping techniques (Sections 7.2.3 or 7.2.4). The returned water should be heavily laden with suspended fines. The water will be discharged to 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- In some cases, the bailer or Watterra[®] foot valve can act as a surge block, flushing water in and out of the well screen as groundwater is removed.
- Repeat the process of surging and pumping/bailing. As development continues, slowly increase the depth of surging to the bottom of the well screen. Surging within the riser portion of the well is neither necessary nor effective.

7.2.2 Bailer

- Tie a string or other cable securely to the bailer. Lower it to the screened or open interval of the monitoring well below the static water level.
- The bailer may be raised and lowered repeatedly within the screened interval to attempt to simulate the action of a surge block by pulling fines through the well screen, and pushing water out into the formation to break down bridging.

- With the bailer full of water, remove it from the well and discharge the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- The Watterra® system (Section 7.2.3) or electric submersible pump (Section 7.2.4) may be used as a complementary development method to the bailer, especially when removal of additional water at a faster rate is beneficial.
- Continue alternately surging and bailing, monitoring the purge water periodically (Section 7.3.1) until development completion criteria are met (Section 7.3.2).

7.2.3 Watterra® system

- Attach high-density polyethylene (HDPE) tubing to the decontaminated Watterra® pump foot valve
- Lower the foot valve and tubing assembly near the bottom of the well.
- Lift and lower the tubing to allow water to enter the Watterra® foot valve and travel up the tubing and discharge the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- The lifting and lowering action of the Watterra® system will cause some surging action to aid in breaking up fine material in the surrounding formation.
- A bailer (Section 7.2.2) may be used as a complementary development method to the Watterra® system, especially during the initial stages of development when a high volume of sediment may be required to be removed.
- An electric submersible pump (Section 7.2.4) may also be used as a complementary development method to the Watterra® system, especially when more volume of water is desired to be pumped or the turbidity criteria cannot be met due to the surging action of the Watterra® system.
- Continue alternately surging and pumping, monitoring the purge water periodically (Section 7.3.1) until well development completion criteria are met (Section 7.3.2).

7.2.4 Electric Submersible Pump

- Attach HDPE tubing to the decontaminated electric submersible pump.
- Lower the pump and tubing assembly near the bottom of the well, at least a few inches above the well total depth.
- Begin pumping, discharging the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- Continue alternately surging and pumping, monitoring the purge water discharge periodically (Section 7.3.1) until well development completion criteria are met (Section 7.3.2).

7.3 Discharge Monitoring

7.3.1 Monitoring the Progress of Development

The progress of the development is evaluated through visual observation of the suspended sediment load and measurement of the turbidity and other parameters in the purged discharge water. As development progresses, the water should become clearer, measured turbidity should decrease, and specific capacity (pumping rate divided by drawdown) should stabilize. Water quality parameters, including DO, conductivity, ORP, pH, temperature, and turbidity may be measured and recorded periodically to determine the progress of development using the criteria outlined in Section 7.3.2 or per the project-specific SAP. Water quality parameters should be measured on each well volume removed.

7.3.2 Completion of Development

The well will be considered developed when the following criteria are met or per the criteria set forth in the project-specific SAP:

- A minimum of three times the standing water volume in a well (to include the well screen and casing plus saturated annulus, assuming 30 percent porosity) is removed.

- Groundwater parameters for three consecutive standing water volumes are within the following:
 - pH – within ± 0.2 units
 - Specific conductivity – within $\pm 3\%$
 - ORP – within ± 10 mV
 - Temperature – within ± 1 degree Celsius
 - Turbidity – at or below 10 nephelometric turbidity units (NTU) or within $\pm 10\%$ if above 10 NTU.
- The sediment thickness remaining within the well is less than 1 percent of the screen length or less than 30 millimeters (0.1 ft) for screens equal to or less than 10 feet long.

Dissolved oxygen (DO) readings may be recorded but DO readings will not be used as development completion criteria because DO may not stabilize.

If the well has slow groundwater recharge and is purged dry, the well will be considered developed when bailed or pumped dry three times in succession and the turbidity has decreased, or per the requirements set forth in the project-specific SAP. Water quality parameters may be recorded if feasible using the flow-through cell.

If any water is added to the well's borehole during development or drilling, three times the volume of water added will also be removed during well development, or per the requirements set forth in the project-specific SAP.

7.4 Development of Wells with Low Yield

Water is the primary mechanism to remove fines and flush water through the gravel pack for effective development. Therefore, development can be a challenge in wells that do not yield sufficient water to recharge when water is removed. However, often these wells are the most in need of development to improve their performance as they are typically installed in low permeability formations with a high content of fines. Development of these wells can improve their yield.

The surging portion of the development can be successfully performed in a well with standing water regardless of its yield. It is the subsequent removal of fine materials that is hindered when insufficient water is recharged to the well. When wells go dry or drawdown significantly during development, development can be performed intermittently, allowing sufficient water to recharge prior conducting the next stage of surging. These intermittent procedures can take place hours or even days apart, depending on project-specific time constraints.

7.5 Wells containing NAPL

Additional care should be taken when planning development of wells that contain NAPL. If the NAPL is flammable, there are health and safety as well as handling issues to consider. If NAPL in excess of a persistent sheen is noted, the recharge rate will be evaluated through hand bailing. In most cases, it is generally preferable to remove NAPL by bailing to the extent practical prior to performing development. Groundwater parameters, excluding turbidity, will not be collected during well development if NAPL or excessive sheen is noticed in the purged water during development to ensure the meter probes are not fouled or destroyed. Well development will be halted.

Development by surging or pumping the well dry can result in the spreading of NAPL vertically in the soil column around the well. These methods can be used, if information exists describing the vertical thickness of the NAPL smear zone around the well, and if the methods do not result in mounding or drawdown that exceeds this thickness. Alternate methods such as bailing may also be used, but any method should not allow the well to be pumped dry or result in significant drawdown that would spread the NAPL vertically.

7.6 Temporary Well Points

For certain projects, temporary well points (TWP) may be installed to collect groundwater samples at a site. Since no sand pack, bentonite chips, or bentonite grout are generally used in the construction of the TWPs, development can proceed as soon as sufficient water has entered the well to static conditions. Due to the small diameter of these wells, generally ¾-inch to 1-inch ID, development will be performed using either a small diameter (0.5-inch) bailer and/or a peristaltic pump with dedicated tubing. The TWPs will have minimal water column and may purge dry during development. However, attempts will be made to remove fines from the well prior to sampling. Purging and sampling may occur as soon as approximately 80% of the static water has re-entered the TWP, or per the requirements set forth in the project-specific SAP.

8.0 Quality Control and Assurance

- 8.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific SAP.
- 8.2 Quality control (QC) requirements are dependent on project-specific sampling objectives. The project-specific SAP will provide requirements for equipment decontamination (frequency and materials) and IDW handling.

9.0 Records, Data Analysis, Calculations

- 9.1 All data and information (e.g., development method used) must be documented on field data sheets (Attachment 1) or within site logbooks with permanent ink. Data recorded may include the following:
- Well Location
 - Weather conditions
 - Date and Time
 - Purge Method
 - Reading/measurements obtained

10.0 Attachments or References

Attachment 1 – Well Development Record

SOP 3-05, *IDW Management*.

SOP 3-06, *Equipment Decontamination*.

<i>Author</i>	<i>Reviewer</i>	<i>Revisions (Technical or Editorial)</i>
Shawn Dolan Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (June 2012)

Attachment 1 Well Development Record



Well/Piezometer Development Record

Well ID:

Client: _____

Project No: _____ Date: _____ Developer: _____

Site Location: _____

Well/Piezometer Data

Well ☐ Piezometer ☐ Diameter _____ Material _____

Measuring Point Description _____ Geology at Screen Interval (if known) _____

Depth to Top of Screen (ft.) _____

Depth to Bottom of Screen (ft.) _____ Time of Water Level Measurement _____

Total Well Depth (ft.) _____ Calculate Purge Volume (gal.) _____

Depth to Static Water Level (ft.) _____ Disposal Method _____

Headspace _____

Original Well Development ☐ Redevelopment ☐ Date of Original Development _____

DEVELOPMENT METHOD

PURGE METHOD

Time	Total Volume Purged (gal.)	Flow Rate (gpm)	Turbidity (NTU)	Color	pH	Temp	Other

ACCEPTANCE CRITERIA (from workplan)

Minimum Purge Volume Required _____ gallons

Maximum Turbidity Allowed _____ NTUs

Stabilization of parameters _____ %

Has required volume been removed

Has required turbidity been reached

Has parameters stabilized

If no or N/A explain below:

Yes No N/A

☐ ☐ ☐
☐ ☐ ☐
☐ ☐ ☐

Signature _____

Date: _____

Soil and Rock Classification

Procedure 3-16

1.0 Purpose and Scope

- 1.1 The purpose of this document is to define the standard operating procedure (SOP) to thoroughly describe the physical characteristics of the sample and classify it according to the Unified Soil Classification System (USCS).
- 1.2 This procedure is the Program-approved professional guidance for work performed by Resolution Consultants under the Comprehensive Long-Term Environmental Action Navy (CLEAN) contract (Contract Number N62470-11-D-8013).
- 1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review. If there are procedures whether it be from Resolution Consultants, state and/or federal that are not addressed in this SOP and are applicable to surface water sampling then those procedures may be added as an appendix to the project specific SAP.
- 1.4 It is fully expected that the procedures outlined in this SOP will be followed. Procedural modifications may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by the Program Quality Manager. Deviations to this SOP will be documented in the field records.

2.0 Safety

- 2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling. All **field sampling personnel** responsible for sampling activities must review the project-specific health and safety plan (HASP) paying particular attention to the control measures planned for the sampling tasks. Conduct preliminary area monitoring to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor and liquid phase through the use of respirators and disposable clothing.
- 2.2 In addition, observe standard health and safety practices according to the project-specific HASP. Suggested minimum protection during well sampling activities includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves, rubberized steel-toed boots, and an American National Standards Institute-standard hard hat. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations, and shall always be available on site.
- 2.3 Daily safety briefs will be conducted at the start of each working day before any work commences. These daily briefs will be facilitated by the **Site Safety Officer (SSO)** or designee to discuss the day's events and any potential health risk areas covering every aspect of the work to be completed. Weather conditions are often part of these discussions. As detailed in the HASP, everyone on the field team has the authority to stop work if an unsafe condition is perceived until the conditions are fully remedied to the satisfaction of the SSO.
- 2.4 The health and safety considerations for the work associated with soil classification include:

- At no time during classification activities are personnel to reach for debris near machinery that is in operation, place any samples in their mouth, or come in contact with the soils/rocks without the use of gloves.
- Stay clear of all moving equipment and be aware of pinch points on machinery. Avoid wearing loose fitting clothing.
- When using cutting tools, cut away from yourself. The use of appropriate, task specific cutting tools is recommended.
- To avoid heat/cold stress as a results of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and in case of extreme cold, wear insulating clothing.

3.0 Terms and Definitions

None.

4.0 Interference

None.

5.0 Training and Qualifications

- 5.1 The **Contract Task Order (CTO) Manager** is responsible for ensuring that the soil and rock classification procedures comply with this procedure. The **CTO Manager** is responsible for ensuring that all personnel involved in soil and rock classification shall have the appropriate education, experience, and training to perform their assigned tasks.
- 5.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.
- 5.3 The **Field Manager** is responsible for ensuring that all project **field personnel** follow these procedures.
- 5.4 Field personnel are responsible for the implementation of this procedure. Minimum qualifications for **field sampling personnel** require that one individual on the field team shall have a minimum of 6 months of experience with soil and rock classification.
- 5.5 The **project geologist** and/or **task manager** is responsible for directly supervising the soil and rock classification procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected. If deviations from the procedure are required because of anomalous field conditions, they must first be approved by the **Program Quality Manager** and then documented in the field logbook and associated report or equivalent document.

6.0 Equipment and Supplies

- 6.1 The following equipment list contains materials which may be needed in carrying out the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.
- Personal protective equipment (PPE) and other safety equipment, as required by the HASP
 - Field log book and pen with indelible ink
 - Boring log

- Munsell Soil Color Chart
- Scoopula, spatula, and/or other small hand tools
- California Sampler
- Hand-held penetrometer

7.0 Calibration or Standardization

None.

8.0 Procedure

8.1 Soil Classification

The basic purpose of the classification of soil is to thoroughly describe the physical characteristics of the sample and to classify it according to an appropriate soil classification system. The USCS was developed so that soils could be described on a common basis by different investigators and serve as a "shorthand" description of soil. A classification of a soil in accordance with the USCS includes not only a group symbol and name, but also a complete word description.

Describing soil on a common basis is essential so that soil described by different site qualified personnel is comparable. Site individuals describing soil as part of site activities *must* use the classification system described herein to provide the most useful geologic database for all present and future subsurface investigations and remedial activities.

The site geologist or other qualified individual shall describe the soil and record the description in a boring log, logbook, and/or electronic field data collection device. The essential items in any written soil description are as follows:

- Classification group name (e.g., silty sand)
- Color, moisture, and odor
- Range of particle sizes and maximum particle size
- Approximate percentage of boulders, cobbles, gravel, sand, and fines
- Plasticity characteristics of the fines
- In-place conditions, such as consistency, density, and structure
- USCS classification symbol

The USCS serves as "shorthand" for classifying soil into 15 basic groups:

GW ¹	Well graded (poorly sorted) gravel (>50 percent gravel, <5percent fines)
GP ¹	Poorly graded (well sorted) gravel (>50percent gravel, <5percent fines)
GM ¹	Silty gravel (>50 percent gravel, >15 percent silt)
GC ¹	Clayey gravel (>50 percent gravel, >15 percent clay)
SW ¹	Well graded (poorly sorted) sand (>50 percent sand, <5 percent fines)
SP ¹	Poorly graded (well sorted) sand (>50 percent sand, <5 percent fines)

¹ If percentage of fine is 5 percent to 15 percent, a dual identification shall be given (e.g., a soil with more than 50 percent poorly sorted gravel and 10 percent clay is designated GW-GC.

SM ¹	Silty sand (>50 percent sand, >15 percent silt)
SC ¹	Clayey sand (>50 percent sand, >15 percent clay)
ML ²	Inorganic, low plasticity silt (slow to rapid dilatancy, low toughness, and plasticity)
CL ²	Inorganic, low plasticity (lean) clay (no or slow dilatancy, medium toughness and plasticity)
MH ²	Inorganic elastic silt (no to slow dilatancy, low to medium toughness and plasticity)
CH ²	Inorganic, high plasticity (fat) clay (no dilatancy, high toughness, and plasticity)
OL	Organic low plasticity silt or organic silty clay
OH	Organic high plasticity clay or silt
PT	Peat and other highly organic soil

Figure 8-1 defines the terminology of the USCS. Flow charts presented in Figure 8-2 and indicate the process for describing soil. The particle size distribution and the plasticity of the fines are the two properties of soil used for classification. In some cases, it may be appropriate to use a borderline classification (e.g., SC/CL) if the soil has been identified as having properties that do not distinctly place the soil into one group.

8.1.1 Estimation of Particle Size Distribution

One of the most important factors in classifying a soil is the estimated percentage of soil constituents in each particle size range. Being proficient in estimating this factor requires extensive practice and frequent checking. The steps involved in determining particle size distribution are listed below:

1. Select a representative sample (approximately 1/2 of a 6-inch long by 2.5-inch diameter sample liner).
2. Remove all particles larger than 3 inches from the sample. Estimate and record the percent by volume of these particles. Only the fraction of the sample smaller than 3 inches is classified.
3. Estimate and record the percentage of dry mass of gravel (less than 3 inches and greater than 1/4 inch).
4. Considering the rest of the sample, estimate, and record the percentage of dry mass of sand particles (about the smallest particle visible to the unaided eye).
5. Estimate and record the percentage of dry mass of fines in the sample (do not attempt to separate silts from clays).
6. Estimate percentages to the nearest 5 percent. If one of the components is present in a quantity considered less than 5 percent, indicate its presence by the term "trace".
7. The percentages of gravel, sand, and fines must add up to 100 percent. "Trace" is not included in the 100 percent total.

8.1.2 Soil Dilatancy, Toughness, and Plasticity

8.1.2.1 Dilatancy

To evaluate dilatancy, follow these procedures:

² If the soil is estimated to have 15 percent to 25 percent sand or gravel, or both, the words "with sand" or "with gravel" (whichever predominates) shall be added to the group name (e.g., clay with sand, CL; or silt with gravel, ML). If the soil is estimated to have 30 percent or more sand or gravel, or both, the words "sandy" or "gravelly" (whichever predominates) shall be added to the group name (e.g., sandy clay, CL). If the percentage of sand is equal to the percent gravel, use "sandy."

1. From the specimen, select enough material to mold into a ball about 1/2 inch (12 millimeters [mm]) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.
2. Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 8-1. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

Table 8-1: Criteria for Describing Dilatancy
















Description	Criteria
None	No visible change in specimen.
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing.
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing.

8.1.2.2 Toughness

Following the completion of the dilatancy test, shape the test specimen into an elongated pat and roll it by hand on a smooth surface or between the palms into a thread about 1/8 inch (3 mm) in diameter. (If the sample is too wet to roll easily, spread it into a thin layer and allow it to lose some water by evaporation.) Fold the sample threads and re-roll repeatedly until the thread crumbles at a diameter of about 1/8 inch. The thread will crumble at a diameter of 1/8 inch when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, lump the pieces together and knead it until the lump crumbles. Note the toughness of the material during kneading. Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 8-2.

Table 8-2: Criteria for Describing Toughness

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft.
Medium	Medium pressure is required to roll the thread near the plastic limit. The thread and the lump have medium stiffness.
High	Considerable pressure is required to roll the thread near the plastic limit. The thread and the lump have very high stiffness.

DEFINITION OF TERMS							
MAJOR DIVISIONS		SYMBOLS		TYPICAL DESCRIPTIONS			
COARSE GRAINED SOILS More Than Half of Material is Larger Than No. 200 Sieve Size	GRAVELS More Than Half of Coarse Fraction is Smaller Than No. 4 Sieve	CLEAN GRAVELS (Less than 6% Fines)		GW	Well graded gravels, gravel-sand mixtures, little or no fines		
				GP	Poorly graded gravels, gravel-sand mixtures, little or no fines		
		GRAVELS With Fines		GM	Silty gravels, gravel-sand-silt mixtures, non-plastic fines		
				GC	Clayey gravels, gravel-sand-clay mixtures, plastic fines		
	SANDS More Than Half of Coarse Fraction is Smaller Than No. 4 Sieve	CLEAN SANDS (Less than 6% Fines)		SW	Well graded sands, gravelly sands, little or no fines		
				SP	Poorly graded sands, gravelly sands, little or no fines		
		SANDS With Fines		SM	Silty sands, sand-silt mixtures, non-plastic fines		
				SC	Clayey sands, sand-clay mixtures, plastic fines		
FINE GRAINED SOILS More Than Half of Material is Smaller Than No. 200 Sieve Size	SILTS AND CLAYS Liquid Limit is Less Than 50%			ML	Inorganic silts, rock flour, fine sandy silts or clays, and clayey silts with non- or slightly-plastic fines		
				CL	Inorganic clays of low to medium plasticity, gravelly clays, silty clays, sandy clays, lean clays		
				OL	Organic silts and organic silty clays of low plasticity		
	SILTS AND CLAYS Liquid Limit is Greater Than 50%			MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts, clayey silt		
				CH	inorganic clays of high plasticity, fat clays		
				OH	Organic clays of medium to high plasticity, organic silts		
			HIGHLY ORGANIC SOILS				PT

GRAIN SIZES							
SILTS AND CLAYS	SAND			GRAVEL		COBBLES	BOULDERS
	FINE	MEDIUM	COARSE	FINE	COARSE		
	200	40	10	4	3/4"	3"	12"
U.S. STANDARD SERIES SIEVE				CLEAR SQUARE SIEVE OPENINGS			

Figure8-1: Unclassified Soil Classification System (USCS)

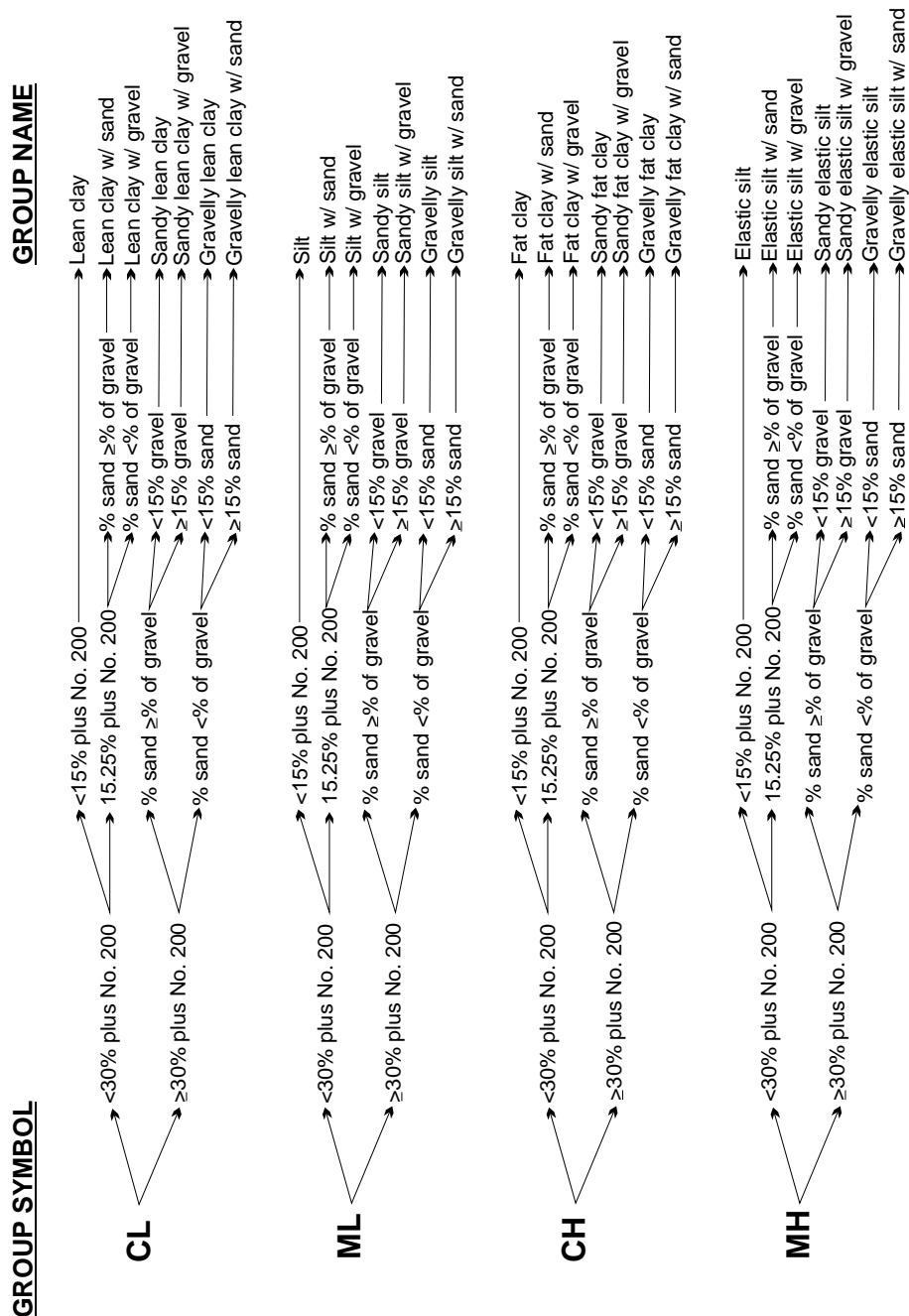


Figure 8-2: Flow Chart for Fine Grain Soil Classification

8.1.2.3 Plasticity

The plasticity of a soil is defined by the ability of the soil to deform without cracking, the range of moisture content over which the soil remains in a plastic state, and the degree of cohesiveness at the plastic limit. The plasticity characteristic of clays and other cohesive materials is defined by the liquid limit and plastic limit. The liquid limit is defined as the soil moisture content at which soil passes from the liquid to the plastic state as moisture is removed. The test for the liquid limit is a laboratory, not a field, analysis.

The plastic limit is the soil moisture content at which a soil passes from the plastic to the semi-solid state as moisture is removed. The plastic limit test can be performed in the field and is indicated by the ability to roll a 1/8-inch (0.125-inch) diameter thread of fines, the time required to roll the thread, and the number of times the thread can be re-rolled when approaching the plastic limit.

The plasticity tests are not based on natural soil moisture content, but on soil that has been thoroughly mixed with water. If a soil sample is too dry in the field, add water prior to performing classification. If a soil sample is too sticky, spread the sample thin and allow it to lose some soil moisture.

Table 8-3 presents the criteria for describing plasticity in the field using the rolled thread method.

Table 8-3: Criteria for Describing Plasticity

Description	Criteria
Non-Plastic	A 1/8-inch thread cannot be rolled.
Low Plasticity	The thread can barely be rolled.
Medium Plasticity	The thread is easy to roll and not much time is required to reach the plastic limit.
High Plasticity	It takes considerable time rolling the thread to reach the plastic limit.

8.1.3 Angularity

The following criteria describe the angularity of the coarse sand and gravel particles:

- **Rounded** particles have smoothly-curved sides and no edges.
- **Subrounded** particles have nearly plane sides, but have well-rounded corners and edges.
- **Subangular** particles are similar to angular, but have somewhat rounded or smooth edges.
- **Angular** particles have sharp edges and relatively plane sides with unpolished surfaces. Freshly broken or crushed rock would be described as angular.

8.1.4 Color, Moisture, and Odor

The natural moisture content of soil is very important. Table 8-4 shows the terms for describing the moisture condition and the criteria for each.

Table 8-4: Soil Moisture Content Qualifiers

Qualifier	Criteria
Dry	Absence of moisture, dry to the touch
Moist	Damp but no visible water
Wet	Visible water, usually soil is below water table

Color is described by hue and chroma using the Munsell Soil Color Chart (Munsell 2000). For uniformity, all site geologists shall utilize this chart for soil classification. Doing so will facilitate correlation of geologic units between boreholes logged by different geologists. The Munsell Color Chart is a small booklet of numbered color chips with names like "5YR 5/6, yellowish-red." Note mottling or banding of colors. It is particularly important to note and describe staining because it may indicate contamination.

In general, wear a respirator if strong organic odors are present. If odors are noted, describe them if they are unusual or suspected to result from contamination. An organic odor may have the distinctive smell of decaying vegetation. Unusual odors may be related to hydrocarbons, solvents, or other chemicals in the subsurface. An organic vapor analyzer may be used to detect the presence of volatile organic contaminants.

8.1.5 **In-Place Conditions**

Describe the conditions of undisturbed soil samples in terms of their density/consistency (i.e., compactness), cementation, and structure utilizing the following guidelines:

8.1.5.1 *Density/Consistency*

Density and consistency describe a physical property that reflects the relative resistance of a soil to penetration. The term “density” is commonly applied to coarse to medium-grained sediments (i.e., gravels, sands), whereas the term “consistency” is normally applied to fine-grained sediments (i.e., silts, clays). There are separate standards of measure for both density and consistency that are used to describe the properties of a soil.

The density or consistency of a soil is determined by observing the number of blows required to drive a 1 3/8-inch (35 mm) diameter split barrel sampler 18 inches using a drive hammer weighing 140 lbs (63.5 kilograms [kg]) dropped over a distance of 30 inches (0.76 meters). Record the number of blows required to penetrate each 6 inches of soil in the field boring log during sampling. The first 6 inches of penetration is considered to be a seating drive; therefore, the blow count associated with this seating drive is recorded, but not used in determining the soil density/consistency. The sum of the number of blows required for the second and third 6 inches of penetration is termed the “standard penetration resistance,” or the “N-value.” The observed number of blow counts must be corrected by an appropriate factor if a different type of sampling device (e.g., Modified California Sampler with liners) is used. For a 2 3/8-inch inner diameter (I.D.) Modified California Sampler equipped with brass or stainless steel liners and penetrating a cohesionless soil (sand/gravel), the N-value from the Modified California Sampler must be divided by 1.43 to provide data that can be compared to the 1 3/8-inch diameter sampler data.

For a cohesive soil (silt/clay), the N-value for the Modified California Sampler should be divided by a factor of 1.13 for comparison with 1 3/8-inch diameter sampler data.

Drive the sampler and record blow counts for each 6-inch increment of penetration until one of the following occurs:

- A total of 50 blows have been applied during any one of the three 6-inch increments; a 50-blow count occurrence shall be termed “refusal” and noted as such on the boring log.
- A total of 150 blows have been applied.
- The sampler is advanced the complete 18 inches without the limiting blow counts occurring, as described above.

If the sampler is driven less than 18 inches, record the number of blows per partial increment on the boring log. If refusal occurs during the first 6 inches of penetration, the number of blows will represent the N-value for this sampling interval. Table 8-5 and Table 8-6 present representative descriptions of soil density/consistency vs. N-values.

Table 8-5: Measuring Soil Density with a California Sampler – Relative Density (Sands, Gravels)

Description	Field Criteria (N-Value)	
	1 3/8 in. ID Sampler	2 in. ID Sampler using 1.43 factor
Very Loose	0–4	0–6
Loose	4–10	6–14
Medium Dense	10–30	14–43
Dense	30–50	43–71
Very Dense	> 50	> 71

Table 8-6: Measuring Soil Density with a California Sampler – Fine Grained Cohesive Soil

Description	Field Criteria (N-Value)	
	1 3/8 in. ID Sampler	2 in. ID Sampler using 1.13 factor
Very Soft	0–2	0–2
Soft	2–4	2–4
Medium Stiff	4–8	4–9
Stiff	8–16	9–18
Very Stiff	16–32	18–36
Hard	> 32	> 36

For undisturbed fine-grained soil samples, it is also possible to measure consistency with a hand-held penetrometer. The measurement is made by placing the tip of the penetrometer against the surface of the soil contained within the sampling liner or Shelby tube, pushing the penetrometer into the soil a distance specified by the penetrometer manufacturer, and recording the pressure resistance reading in pounds per square foot (psf). The values are as follows (Table 8-7):

Table 8-7: Measuring Soil Consistency with a Hand-Held Penetrometer

Description	Pocket Penetrometer Reading (psf)
Very Soft	0–250
Soft	250–500
Medium Stiff	500–1000
Stiff	1000–2000
Very Stiff	2000–4000
Hard	>4000

Consistency can also be estimated using thumb pressure using Table 8-8.

Table 8-8: Measuring Soil Consistency Using Thumb Pressure

Description	Criteria
Very Soft	Thumb will penetrate soil more than 1 inch (25 mm)
Soft	Thumb will penetrate soil about 1 inch (25 mm)
Firm	Thumb will penetrate soil about 1/4 inch (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very Hard	Thumbnail will not indent soil

8.1.5.2 *Cementation*

Cementation is used to describe the friability of a soil. Cements are chemical precipitates that provide important information as to conditions that prevailed at the time of deposition, or conversely, diagenetic effects that occurred following deposition. Seven types of chemical cements are recognized by Folk (1980). They are as follows:

- Quartz – siliceous
- Chert – chert-cemented or chalcedonic
- Opal – opaline
- Carbonate – calcitic, dolomitic, sideritic (if in doubt, calcareous should be used)
- Iron oxides – hematitic, limonitic (if in doubt, ferruginous should be used)
- Clay minerals – if the clay minerals are detrital or have formed by recrystallization of a previous clay matrix, they are not considered to be a cement. Only if they are chemical precipitates, filling previous pore space (usually in the form of accordion-like stacks or fringing radial crusts) should they be included as “kaolin-cemented,” “chlorite-cemented,” etc.
- Miscellaneous minerals – pyritic, collophane-cemented, glauconite-cemented, gypsiferous, anhydrite-cemented, baritic, feldspar-cemented, etc.

The degree of cementation of a soil is determined qualitatively by utilizing finger pressure on the soil in one of the sample liners to disrupt the gross soil fabric. The three cementation descriptors are as follows:

- Weak – friable; crumbles or breaks with handling or slight finger pressure
- Moderate – friable; crumbles or breaks with considerable finger pressure
- Strong – not friable; will not crumble or break with finger pressure

8.1.5.3 *Structure*

This variable is used to qualitatively describe physical characteristics of soil that are important to incorporate into hydrogeological and/or geotechnical descriptions of soil at a site. Appropriate soil structure descriptors are as follows:

- Granular – spherically shaped aggregates with faces that do not accommodate adjoining faces
- Stratified – alternating layers of varying material or color with layers at least 6 mm (1/4 inch) thick; note thickness
- Laminated – alternating layers of varying material or color with layers less than 6 mm (1/4 inch) thick; note thickness
- Blocky – cohesive soil that can be broken down into small angular or subangular lumps that resist further breakdown
- Lensed – inclusion of a small pocket of different soil, such as small lenses of sand, should be described as homogeneous if it is not stratified, laminated, fissured, or blocky. If lenses of different soil are present, the soil being described can be termed homogeneous if the description of the lenses is included
- Prismatic or Columnar – particles arranged about a vertical line, ped is bounded by planar, vertical faces that accommodate adjoining faces; prismatic has a flat top; columnar has a rounded top
- Platy – particles are arranged about a horizontal plane

8.1.5.4 *Other Features*

- Mottled – soil that appears to consist of material of two or more colors in blotchy distribution
- Fissured – breaks along definite planes of fracture with little resistance to fracturing (determined by applying moderate pressure to sample using thumb and index finger)
- Slickensided – fracture planes appear polished or glossy, sometimes striated (parallel grooves or scratches)

8.1.6 **Development of Soil Description**

Develop standard soil descriptions according to the following examples. There are three principal categories under which all soil can be classified. They are described below.

8.1.6.1 *Coarse-grained Soil*

Coarse-grained soil is divided into sands and gravels. A soil is classified as a sand if over 50 percent of the coarse fraction is “sand-sized.” It is classified as a gravel if over 50 percent of the coarse fraction is composed of “gravel-sized” particles.

The written description of a coarse-grained soil shall contain, in order of appearance: Typical name including the second highest percentage constituent as an adjective, if applicable (underlined); grain size of coarse fraction; Munsell color and color number; moisture content; relative density; sorting; angularity; other features, such as stratification (sedimentary structures) and cementation, possible formational name, primary USCS classification, secondary USCS classification (when necessary), and approximate percentages of minor constituents (i.e., sand, gravel, shell fragments, rip-up clasts) in parentheses.

Example: POORLY-SORTED SAND WITH SILT, medium- to coarse-grained, light olive gray, 5Y 6/2, saturated, loose, poorly sorted, subrounded clasts, SW/SM (minor silt with approximately 20 percent coarse-grained sand-sized shell fragments, and 80 percent medium-grained quartz sand, and 5 percent to 15 percent ML).

8.1.6.2 *Fine-grained Soil*

Fine-grained soil is further subdivided into clays and silts according to its plasticity. Clays are rather plastic, while silts have little or no plasticity.

The written description of a fine-grained soil should contain, in order of appearance: Typical name including the second highest percentage constituent as an adjective, if applicable (underlined); Munsell color; moisture content; consistency; plasticity; other features, such as stratification, possible formation name, primary USCS classification, secondary USCS classification (when necessary), and the percentage of minor constituents in parentheses.

Example: SANDY LEAN CLAY, dusky red, 2.5 YR 3/2, moist, firm, moderately plastic, thinly laminated, CL (70 percent fines, 30 percent sand, with minor amounts of disarticulated bivalves [about 5 percent]).

8.1.6.3 *Organic Soil*

For highly organic soil, describe the types of organic materials present as well as the type of soil constituents present using the methods described above. Identify the soil as an organic soil, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soil usually has a dark brown to black color and may have an organic odor. Often, organic soils will change color, (e.g., from black to brown) when exposed to air. Some organic soils will lighten in color significantly when air-dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

8.2 Example: ORGANIC CLAY, black, 2.5Y, 2.5/1, wet, soft, low plasticity, organic odor, OL (100 percent fines), weak reaction to HCl.

8.3 **Rock Classification**

The purpose of rock classification is to thoroughly describe the physical and mineralogical characteristics of a specimen and to classify it according to an established system. The generalized rock classification system described below was developed because, unlike the USCS for soils, there is no universally accepted rock classification system. In some instances, a more detailed and thorough rock classification system may be appropriate. Any modifications to this classification system, or the use of an alternate classification system should be considered during preparation of the site work plan. Both the CTO Manager and the QA Manager or Technical Director must approve any modifications to this classification system, or the use of another classification system.

Describing rock specimens on a common basis is essential so that rocks described by different site geologists are comparable. Site geologists describing rock specimens as a part of investigative activities must use the classification system described herein, or if necessary, another more detailed classification system. Use of a common classification system provides the most useful geologic database for all present and future subsurface investigations and remedial activities.

In order to provide a more consistent rock classification between geologists, a rock classification template has been designated as shown in **Error! Reference source not found.**. The template includes classification of rocks by origin and mineralogical composition. When classifying rocks, all site geologists shall use this template.

The site geologist shall describe the rock specimen and record the description in a boring log or logbook. The items essential for classification include (i.e., metamorphic foliated):

- Classification Name (i.e., schist)
- Color
- Mineralogical composition and percent
- Texture/Grain size (i.e., fine-grained, pegmatitic, aphyritic, glassy)
- Structure (i.e., foliated, fractured, lenticular)
- Rock Quality Designation (sum of all core pieces greater than two times the diameter of the core divided by the total length of the core run, expressed as a percentage)
- Classification symbol (i.e., MF)

Example: Metamorphic foliated schist: Olive gray, 5Y, 3/2, Garnet 25 percent, Quartz 45 percent, Chlorite 15 percent, Tourmaline 15 percent, Fine-grained with Pegmatite garnet, highly foliated, slightly wavy, MF.

9.0 Quality Control and Assurance

None


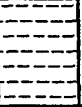

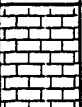


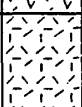

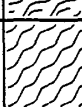
DEFINITION OF TERMS					
PRIMARY DIVISIONS			SYMBOLS		SECONDARY DIVISIONS
SEDIMENTARY ROCKS	Clastic Sediments	CONGLOMERATE		CG	Coarse-grained Clastic Sedimentary Rock types including: Conglomerates and Breccias
		SANDSTONE		SS	Clastic Sedimentary Rock types including: Sandstone, Arkose and Greywacke
		SHALE		SH	Fine-grained Clastic Sedimentary Rock types including: Shale, Siltstone, Mudstone and Claystone
	Chemical Precipitates	CARBONATES		LS	Chemical Precipitates including: Limestone, Crystalline Limestone, Fossiliferous Limestone Micrite and Dolomite
		EVAPORITES		EV	Evaporites including: Anhydrite, Gypsum, Halite, Travertine and Caliche
IGNEOUS ROCKS	EXTRUSIVE (Volcanic)			IE	Volcanic Rock types including: Basalt, Andesite, Rhyolite, Volcanic Tuff, and Volcanic Breccia
	INTRUSIVE (Plutonic)			II	Plutonic Rock types including: Granite, Diorite and Gabbro
METAMORPHIC ROCKS	FOLIATED			MF	Foliated Rock types including: Slate, Phyllite, Schist and Gneiss
	NON-FOLIATED			MN	Non-foliated Rock types including: Metaconglomerate, Quartzite and Marble

Figure 8-4: Rock Classification System

10.0 Data and Records Management

- 10.1 Document soil classification information collected during soil sampling onto the field boring logs, field trench logs, and into the field notebook. Copies of this information shall be sent to the **CTO Manager** for the project files.
- 10.2 Field notes will be kept during coring activities in accordance with SOP 3-03 – Recordkeeping, Sample Labeling, and Chain of Custody. The information pertinent to soil classification activities includes chronology of events, sample locations (x,y,z), time/date, sampler name, methods (including type of core liner/barrel, if applicable), sampler penetration and acceptability, sample observations, and the times and type of equipment decontamination. Deviations to the procedures detailed in the SOP should be recorded in the field logbook.

11.0 Attachments or References

American Society for Testing and Materials (ASTM). 2000. *Standard Practice for Description and Identification of Soils (Visual, Manual Procedure)*. D 2488-00. West Conshohocken, PA.

Birkeland, Peter W. 1984. *Soils and Geomorphology*. 3rd ed. New York: Oxford University Press.

Compton, Robert R. 1985. *Geology in the Field*. Hoboken, NJ: John Wiley & Sons, Inc.

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Department of the Navy (DON). 2007. *Navy Environmental and Natural Resources Program Manual*. OPNAV Instruction 5090.1c. October.

Folk, Robert L. 1980. *Petrology of Sedimentary Rocks*. Austin, TX: Hemphill Publishing Company.

Huang, Walter T. 1962. *Petrology*. New York: McGraw-Hill Book Company.

McCarthy, David F. 2005. *Essentials of Soil Mechanics and Foundations: Basic Geotechnics*. 7th Ed. Indianapolis, IN: Prentice Hall. July.

Munsell Color Company (Munsell). 2000. *Munsell Soil Color Chart*, (Revised). Baltimore.

Pettijohn, F.J. 1957. *Sedimentary Rocks*. 2nd Edition. New York: Harper and Brothers.

Rahn, Perry H. 1996. *Engineering Geology*. 2nd Edition. Indianapolis, IN: Prentice Hall. August

Author	Reviewer	Revisions (Technical or Editorial)
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Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Sample and Evidence Management**

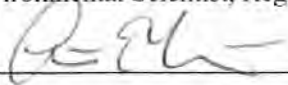
Effective Date: January 29, 2013

Number: SESDPROC-005-R2

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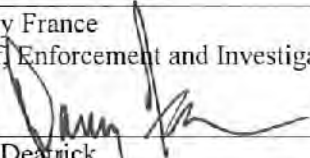
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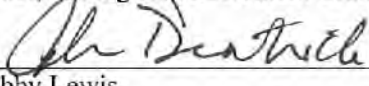
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Date: 1/23/13

Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
<p>SESDPROC-005-R2, <i>Sample and Evidence Management</i>, replaces SESDPROC-005-R1</p> <p>General: Corrected any typographical, grammatical, and/or editorial errors.</p> <p>General: Replaced all references to FORMS with the generic term sample custody software program. The specific sections are listed below: Section 3.3 Section 3.4 Section 4.2 Section 5</p> <p>Title Page: Changed the EIB Branch Chief from Antonio Quinones to Danny France. Changed the EAB Branch Chief from Bill Cosgrove to John Deatrick (Acting). Changed the Field Quality Manager from Laura Ackerman to Bobby Lewis.</p> <p>Revision History: In the last sentence, changed Field Quality Manager to Document Control Coordinator.</p> <p>Section 1.2: Added the following statement - Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.</p> <p>Section 1.3: Changed requirement so that the DCC is responsible for ensuring the most recent version of the procedure is placed on the SESD LAN and for maintaining records of review conducted prior to its issuance. Deleted reference to the H: drive.</p> <p>Section 2.2 Changed requirement for comments on sample label to make comments optional.</p> <p>Section 2.2.2 Removed requirement for printed copies of photographs in the official file.</p> <p>Section 3.3 Removed different custody requirements for criminal investigations so that all projects are treated consistently.</p> <p>Section 3.4 Removed statement in the fourth paragraph regarding retention of paper air bills in the official project file to reduce unnecessary paperwork. The air bill number or shipment tracking number is recorded on the chain of custody.</p>	<p>January 29, 2013</p>

<p>SESDPROC-005-R1, <i>Sample and Evidence Management</i>, replaces SESDPROC-005-R0</p> <p>General Updated referenced procedures to reflect most recent version.</p> <p>Replaced “shall” with “will”.</p> <p>Cover Page: Changed title for Antonio Quinones from Environmental Investigation Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove’s title from Acting Chief to Chief.</p> <p>Section 1.3 Updated information to reflect that procedure is located on the H: drive of the LAN.</p> <p>Section 1.4 Added reference for the SESD Operating Procedure for Control of Records. Alphabetized and revised the referencing style for consistency.</p> <p>Section 2.2.3 Added that Confidential Business Information will be handled in accordance with SESD Operating Procedure for Control of Records.</p>	<p>November 1, 2007</p>
<p>SESDPROC-005-R0, <i>Sample and Evidence Management</i>, Original Issue</p>	<p>February 05, 2007</p>

TABLE OF CONTENTS

1	General Information.....	5
1.1	Purpose.....	5
1.2	Scope/Application.....	5
1.3	Documentation/Verification	5
1.4	References	5
2	Sample and Evidence Identification.....	6
2.1	Introduction	6
2.2	Sample and Evidence Identification Procedures.....	7
2.2.1	<i>Sample Identification</i>	7
2.2.2	<i>Photograph, Digital Still Image and Video Identification</i>	7
2.2.3	<i>Identification of Physical Evidence</i>	8
3	Chain-of-Custody Procedures	10
3.1	Introduction	10
3.2	Sample Custody	10
3.3	Documentation of Chain-of-Custody.....	10
3.4	Transfer of Custody with Shipment	12
4	Receipt for Samples Form (CERCLA/RCRA/TSCA).....	14
4.1	Introduction	14
4.2	Receipt for Samples Form	14
5	Sample Custody Management Software.....	16

1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed by SESD field investigators when handling and managing samples and other types of evidence after their collection and during delivery to the laboratory.

1.2 Scope/Application

The procedures contained in this document are to be used by field investigators when handling and managing samples and other evidence collected to support SESD field investigations. On the occasion that SESD field investigators determine that any of the procedures described in this section are either inappropriate, inadequate or impractical and that another procedure must be used, the variant procedure will be documented in the field log book, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version

SESD Operating Procedure for Packing, Marking, Labeling and Shipping of Environmental and Waste Samples, SESDPROC-209, Most Recent Version

USEPA Region 4 Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM), November 2001

USEPA Digital Camera Guidance for EPA Civil Inspections and Investigations, July 2006

2 Sample and Evidence Identification

2.1 Introduction

Sample identification, chain-of-custody records, receipt for sample records and other field records will be legibly recorded with waterproof, non-erasable ink, unless otherwise specified. If errors are made in any of these documents, corrections will be made by crossing a single line through the error and entering the correct information. All corrections must be initialed and dated. If possible, all corrections should be made by the individual making the error.

Following are definitions of terms used in this section:

Field Investigator

Any individual who performs or conducts field sampling, observation and/or measurement activities in support of field investigations

Project Leader

The individual with overall responsibility for conducting a specific field investigation in accordance with this procedure

Field Sample Custodian

Individual responsible for identifying the sample containers and maintaining custody of the samples and the Chain-of-Custody Record

Sample Team Leader

An individual designated by the project leader to be present during and responsible for all activities related to the collection of samples by a specific sampling team

Sampler

The individual responsible for the actual collection of a sample

Transferee

Any individual who receives custody of samples subsequent to release by the field sample custodian

Laboratory Sample Custodian

Individual responsible for accepting custody of samples from the field sample custodian or a transferee

One individual may fulfill more than one of the roles described above.

2.2 Sample and Evidence Identification Procedures

2.2.1 Sample Identification

The method of sample identification used depends on the type of sample collected. Field measurement samples are those collected for specific field analysis or measurement where the data are recorded directly in bound field logbooks or on the Chain-of-Custody Record. Examples of field measurements and analyses include XRF, pH, temperature, dissolved oxygen and conductivity. Samples collected for laboratory analysis will be identified by using a stick-on label or a tag which is attached to the sample container. In some cases such as biological samples, the label or tag may have to be affixed to a bag containing the sample. If a sample tag is used, the sample should be placed in a bag, then the sample and the tag will be placed in a second bag.

The following information will be included on the sample label or tag using waterproof, non-erasable ink:

- Project number;
- Field identification or sample station number;
- Date and time of sample collection;
- Designation of the sample as a grab or composite;
- Whether the sample is preserved or unpreserved;
- The general types of analyses to be performed.

Other information such as readily detectable or identifiable odor, color, or known toxic properties may be added as deemed necessary by the project leader or sample custodian.

2.2.2 Photograph, Digital Still Image and Video Identification

Photographs and Digital Still Images

When photographs or digital images are taken for purposes of documenting and supporting a field investigation, a record of each exposure or image will be kept in a bound field logbook. The following information will be recorded in the logbook:

- An accurate description of what the photograph or image shows, including orientation, if appropriate;

- The date and time that the photograph or image was taken;
- The name of the individual who took the photograph or digital image.

When photographs are taken with a film camera, the film should be developed with the negatives supplied uncut, if possible. The identifying information that was recorded in the field logbook will be entered on the back of the prints.

When digital images are obtained during a field investigation, an electronic copy of the unaltered investigation-related images will be placed in the official files. If deemed necessary due to project requirements, a printed copy of the original photographs may be placed in the official file. For enforcement cases, it is imperative that the individual who took the image be identified in the field logbook in the event their testimony is required.

Video

When a video recording is used as evidence in an enforcement case, the following information should be recorded in a bound field logbook:

- The date and time that the video was recorded;
- A brief description of the subject of the video;
- The person recording the video.

An audio record may also be included in the video tape with the above logistical information, as well as a narrated description of the video record.

A label will be placed on the video media with the appropriate identifying information (i.e., project name, project number, date, location etc.). In the event testimony regarding a video recording is required for an enforcement case, one individual should be responsible for recording the video for each case. The original, unaltered recording will be placed in the official files.

2.2.3 Identification of Physical Evidence

Physical evidence, other than samples, will be identified, when possible, by recording the necessary information on the evidence. When samples are collected from vessels or containers which can be moved (drums for example), the vessel or container should be marked with the field identification or sample station number for future identification. The vessel or container may be labeled with an indelible marker (e.g., paint stick or spray paint). The vessel or container need not be marked if it already has a unique marking; however, these markings will be

recorded in the bound field logbooks. In addition, it is suggested that photographs of any physical evidence (markings, etc.) be taken and the necessary information recorded in the field logbook.

Occasionally, it is necessary to obtain copies of recorder and/or instrument charts from facility owned analytical equipment, flow recorders, etc., during field investigations and inspections. A unique identifier will be recorded on the document with that information as well as the following recorded in the logbook:

- Starting and ending time(s) and date(s) for the chart;
- An instantaneous measurement of the media being measured by the recorder will be taken and entered at the appropriate location on the chart along with the date and time of the measurement; and
- A description of the location being monitored and other information required to interpret the data such as type of flow device, chart units, factors, etc.

The field investigator will indicate who the chart (or copy of the chart) was received from and enter the date and time, as well as the field investigator's initials.

Documents such as technical reports, laboratory reports, etc., should be marked with the field investigator's signature, the date, the number of pages and from whom they were received. Documents that are claimed by a facility to be "confidential" and, therefore, potentially subject to the Confidential Business Information requirements, will be handled in accordance with SESD Operating Procedure for Control of Records (SESDPROC-002).

3 Chain-of-Custody Procedures

3.1 Introduction

Chain-of-custody procedures are comprised of the following elements: 1) maintaining custody of samples or other evidence, and 2) documentation of the chain-of-custody for evidence. To document chain-of-custody, an accurate record must be maintained to trace the possession of each sample, or other evidence, from the moment of collection to its introduction into evidence.

3.2 Sample Custody

A sample or other physical evidence is in custody if:

- It is in the actual possession of an investigator;
- It is in the view of an investigator, after being in their physical possession;
- It was in the physical possession of an investigator and then they secured it to prevent tampering; and/or
- It is placed in a designated secure area.

3.3 Documentation of Chain-of-Custody

The following are used to identify and demonstrate how sample integrity is maintained and custody is ensured.

Sample Identification

A stick-on sample label or a tag should be completed for each sample container using waterproof, non-erasable ink as specified in Section 2.2.1.

Sample Seals

If appropriate, samples should be sealed as soon as possible following collection using a custody seal with EPA identification. The sample custodian or project leader will write the date and their initials on the seal. The use of custody seals may be waived if field investigators keep the samples in their custody as defined in Section 3.2, from the time of collection until the samples are delivered to the laboratory analyzing the samples.

Field Sample Custodian

The field sample custodian is the person designated by the project leader to receive and manage custody of samples while in the field, including labeling and custody sealing.

Chain-of-Custody Record

The field Chain-Of-Custody record is used to document the custody of all samples or other physical evidence collected and maintained by investigators. All physical evidence or samples will be accompanied by a Chain-Of-Custody Record. This form may be generated by sample custody management software (Section 5) or it may be a pre-printed multi-sheet carbonless form for hand entry of required information. The Chain-Of-Custody Record documents transfer of custody of samples from the sample custodian to another person, to the laboratory or other organizational elements. The Chain-of-Custody Record will not be used to document the collection of split samples where there is a legal requirement to provide a receipt for samples (see Section 4, Receipt for Samples Form (CERCLA/RCRA/TSCA)). The Chain-Of-Custody Record also serves as a sample logging mechanism for the laboratory sample custodian. A separate Chain-of-Custody Record should be used for each final destination or laboratory used during the investigation.

All information necessary to fully and completely document the sample collection and required analyses must be recorded in the appropriate spaces to complete the field Chain-Of-Custody Record. The following requirements apply to Chain-Of-Custody records generated by either sample custody management software or by hand entry on pre-printed forms:

- All sampling team leaders must sign in the designated signature block.
- One sample should be entered on each line and not be split among multiple lines.
- If multiple sampling teams are collecting samples, the sampling team leader's name should be clearly indicated for each sample.
- The total number of sample containers for each sample must be listed in the appropriate column. Required analyses should be entered in the appropriate location on the Chain-of-Custody Record.
- The field sample custodian, project leader or other designee, and subsequent transferee(s) should document the transfer of the samples listed on the Chain-of-Custody Record. Both the person relinquishing the samples and the person receiving them must sign the form. The date and time that this occurs should be documented in the proper space on the Chain-of-Custody Record. The exception to this requirement would be when packaged samples are shipped with a common carrier. Even though the common carrier accepts the samples for shipment, they do not sign the Chain-of-Custody Record as having received the samples.
- The last person receiving the samples or evidence will be the laboratory sample custodian or their designee(s).

The Chain-of-Custody Record is a uniquely identified document. Once the Record is completed, it becomes an accountable document and must be maintained in the project file. The suitability of any other form for chain-of-custody should be evaluated based upon its inclusion of all of the above information in a legible format.

If chain-of-custody is required for documents received during investigations, the documents should be placed in large envelopes, and the contents should be noted on the envelope. The envelope will be sealed and an EPA custody seal placed on the envelope such that it cannot be opened without breaking the seal. A Chain-Of-Custody Record will be maintained for the envelope. Any time the EPA seal is broken, that fact will be noted on the Chain-Of-Custody Record and a new seal affixed, as previously described in this section.

Physical evidence such as video tapes or other small items will be placed in an evidence bag or envelope and an EPA custody seal should be affixed so that they cannot be opened without breaking the seal. A Chain-Of-Custody Record will be maintained for these items. Any time the EPA seal is broken, that fact will be noted on the Chain-of-Custody Record and a new seal affixed.

EPA custody seals can be used to maintain custody of other items when necessary by using similar procedures as those previously outlined in this section.

Samples should not be accepted from other sources unless the sample collection procedures used are known to be acceptable, can be documented and the sample chain-of-custody can be established. If such samples are accepted, a standard sample label containing all relevant information and the Chain-Of-Custody Record will be completed for each set of samples.

3.4 Transfer of Custody with Shipment

Transfer of custody is accomplished by the following:

- Samples will be properly packaged for shipment in accordance with the procedures outlined in SESD Operating Procedure for Packing, Marking, Labeling and Shipping of Environmental and Waste Samples (SESDPROC-209).
- All samples will be accompanied by the laboratory copy of the Chain-Of-Custody Record. If pre-printed forms are used, the white and pink sheets will be sent. If sample custody management software is used to generate the Chain-Of-Custody Record, the laboratory copy is identified with an "L" in the upper right corner. If multiple coolers are needed for shipment to a particular laboratory, the laboratory copy of the Chain-Of-Custody Record for the entire shipment is placed in a sealed plastic bag in one of the coolers. When shipping samples via common carrier, the "Relinquished By" box should be filled in; however, the "Received By" box should be left blank. The laboratory sample custodian is responsible for receiving custody of the samples and will fill in the "Received By" section of the Chain-of-

Custody Record. One copy of the Record will be provided to and retained by the project leader. After samples have been received and accepted by the laboratory, a copy of the Chain-of-Custody Record, with ASB sample identification numbers, will be transmitted to the project leader. This copy will become a part of the project file.

- If sent by mail, the package will be registered with return receipt requested. If sent by common carrier, an Air Bill should be used. The Air Bill number, shipment tracking number or registered mail serial number will be recorded in the remarks section of the Chain-Of-Custody Record.

4 Receipt for Samples Form (CERCLA/RCRA/TSCA)

4.1 Introduction

Section 3007 of the Resource Conservation and Recovery Act (RCRA) of 1976 and Section 104 of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or Superfund) of 1980 require that a "receipt" for all facility samples collected during inspections and investigations be given to the owner/operator of each facility before the field investigator departs the premises. The Toxic Substances Control Act (TSCA) contains similar provisions. The laws do not require that homeowners or other off-site property owners be given this form.

4.2 Receipt for Samples Form

If necessary, a Receipt for Samples form, using either the pre-printed form or one generated by sample custody management software, is to be used to satisfy the receipt for samples provisions of RCRA, CERCLA and TSCA. The form also documents that split samples were offered and either "Received" or "Declined" by the owner/operator of the facility or site being investigated (if a sample is split with a facility, state regulatory agency or other party representative, the recipient should be provided (if enough sample is available) with an equal weight or volume of sample). All information must be supplied in the indicated spaces to complete the Receipt for Samples form.

- The sampler(s) must sign the form in the indicated location
- Each sample collected from the facility or site must be documented in the sample record portion of the form. The sample station number, date and time of sample collection, composite or grab sample designation, whether or not split samples were collected (yes or no should be entered under the split sample column), a brief description of each sampling location and the total number of sample containers for each sample must be entered.
- The bottom of the form is used to document the site operator's acceptance or rejection of split samples. The project leader must sign and complete the information in the "Split Samples Transferred By" section (date and time must be entered). If split samples were not collected, the project leader should initial and place a single line through "Split Samples Transferred By" in this section. The operator of the site must indicate whether split samples were received or declined and sign the form. The operator must give their title, telephone number and the date and time they signed the form. If the operator refuses to sign the form, the sampler(s) should note this fact in the operator's signature block and initial this entry.

The Receipt for Samples form is an accountable document after it is completed. A copy of the form is to be given to the facility or site owner/operator. The original form must be maintained in the project files.

5 Sample Custody Management Software

The container labels and the Chain-of-Custody record should be generated using a sample custody management software to streamline the documentation required by SEDS and/or the Contract Laboratory Program (CLP) for sample identification and chain-of-custody. When possible, the sample custody management software should be used during all field investigations. Once the appropriate information is entered into the computer, the software will generate stick-on labels for the sample containers and will generate sample receipt forms and chain-of-custody records for the appropriate laboratory. The advantages to this system include faster processing of samples and increased accuracy. Accuracy is increased because the information is entered only once, and consequently, consistent for the bottle labels, sample receipt forms and chain-of-custody records. .

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: Logbooks

Effective Date: May 30, 2013

Number: SESDPROC-010-R5

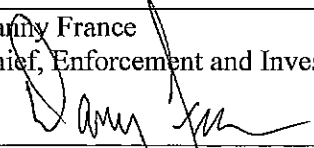
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Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the SESD Document Control Coordinator on the SESD local area network (LAN).

History	Effective Date
<p>SESDPROC-010-R5, <i>Logbooks</i>, replaces SESDPROC-010-R4</p> <p>General: Corrected any typographical, grammatical and/or editorial errors.</p> <p>Title Page: Changed author from Liza Montalvo to Hunter Johnson. Changed Enforcement and Investigation Branch Chief from Archie Lee to Danny France. Changed Ecological Assessment Branch Chief from Bill Cosgrove to John Deatrick. Changed Field Quality Manager from Liza Montalvo to Bobby Lewis.</p> <p>Revision History: Changes were made to reflect the current practice of only including the most recent changes in the revision history</p> <p>Section 1.2: Added the following statement: "Requirements for SESD records, which include field logbooks, are outlined in the SESD Operating Procedure for Control of Records (SESDPROC-002)."</p> <p>Section 1.4: Updated references</p> <p>Section 2.1: Added "bound" to the first sentence.</p> <p>Replaced the last sentence of the first paragraph with the following language: "All pertinent field activity information will be recorded contemporaneously when observed or collected to prevent a loss of information."</p> <p>Added the second paragraph partially comprised of language from the omitted paragraph from Section 2.2.</p> <p>Section2.2: Omitted the first paragraph.</p> <p>Added the following language to the first line (formerly the second paragraph): "The following requirements apply to all logbooks:"</p> <p>Added Items 1 and 2 and omitted Item 4. Renumbered items as appropriate.</p> <p>Added "End of Notes" to the second sentence in item 6 (formerly item 5). Also added the last sentence which states: "Field investigators will draw a diagonal line through blank or unused portions of pages/forms"</p>	<p>May 30, 2013</p>

that are located prior to the “End of Notes” entry and initial them.” Added “and SESD-generated forms” to Item 7 (formerly item 6).	
SESCPROC-010-R4, <i>Logbooks</i> , replaces SESDPROC-010-R3	October 8, 2010
SESCPROC-010-R3, <i>Logbooks</i> , replaces SESDPROC-010-R2	November 1, 2007
SESCPROC-010-R2, <i>Logbooks</i> , Replaces SESDPROC-010-R1	September 25, 2007
SESDPROC-010-R1, <i>Logbooks</i> , Replaces <i>Field Records (Logbooks)</i> SESDPROC-010-R0	August 10, 2007
SESDPROC-010-R0, Field Records (Logbooks), Original Issue	February 5, 2007

TABLE OF CONTENTS

1	General Information	5
1.1	Purpose.....	5
1.2	Scope/Application	5
1.3	Documentation/Verification.....	5
1.4	References	5
2	Field Records and Documentation Procedures.....	6
2.1	General.....	6
2.2	Field Data Integrity and Accountability	6
2.3	Logbook Entry Information	7
2.3.1	<i>General Information Required in All Logbooks.....</i>	<i>7</i>
2.3.2	<i>Information Required for Sample Collection</i>	<i>7</i>
2.3.3	<i>Information Required for Field Measurements</i>	<i>8</i>
2.3.4	<i>Additional Information for Inclusion</i>	<i>9</i>

1 General Information

1.1 Purpose

This procedure is to be used by field investigators when documenting pertinent and factual information in logbooks related to field investigations involving sampling and measurement procedures and/or other data collection events.

1.2 Scope/Application

This document describes the various types of information that should be included in the field logbooks used to document field investigations conducted by SESD. Requirements for SESD records, which include field logbooks, are outlined in the SESD Operating Procedure for Control of Records (SESDPROC-002). Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

The procedures found within this document were prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version

SESD Field Branches Quality Management Plan, SESDPLAN-001, Most Recent Version

2 Field Records and Documentation Procedures

2.1 General

Dedicated bound logbooks will be used for field data collection including but not limited to sampling, measurements and observations. Logbook entries should be objective, factual, and free of personal feelings or other terminology which might prove inappropriate. All pertinent field activity information will be recorded contemporaneously when observed or collected to prevent a loss of information.

To facilitate accurate and complete documentation of field sampling and measurement activities, SESD-generated forms may be used. In order to be utilized, SESD-generated forms must be bound prior to use and adhere to all requirements outlined in this procedure. In cases where unbound pages/forms are necessary due to project requirements or practicality, approval must be obtained from both the Field Quality Manager and Management. In these cases, the Field Quality Manager and Management will dictate the steps that will be taken to ensure credibility, traceability and defensibility of information collected.

Any deviations from the quality assurance project plan that occur while in the field will be noted in the logbook(s). Logbook entries that may be considered privileged or confidential information will be handled in accordance with the relevant sections of SESD Operating Procedure for Control of Records (SESDPROC-002). The logbooks will be placed in the SESD project file upon transmittal of the final report to the project requestor.

2.2 Field Data Integrity and Accountability

The following requirements apply to **all logbooks**:

1. The project's unique identifier (unique identification number(s)) will be included on each page.
2. Field personnel will date and number each page. Numbering will be conducted by utilizing a format that incorporates both the current page number and the total number of pages (e.g. "page x of y" or "x/y", where "x" is the current page number and "y" is the total number of pages).
3. Observations, data and calculations will be recorded at the time they are made.
4. Unless prohibited by environmental conditions, pens with permanent ink will be used to record all data. When environmental conditions do not make it feasible to use permanent ink, entries should be made using a non-smear lead pencil (e.g., 2H or 3H). Upon returning from the field, the project leader will photocopy the penciled section of the logbook and certify, in writing, that the photocopied record is a true copy of the original logbook entry. The photocopy will be included in the project file.

5. Entries will be legible and contain pertinent, accurate and inclusive documentation of project activities.
6. Upon completion of the field investigation, the end of project entries in the logbook and/or bound forms will be clearly indicated. This may be accomplished by noting “End” or “End of Notes” on the last page of notes and dating and initialing the notation. Field investigators will draw a diagonal line through blank or unused portions of pages/forms that are located prior to the “End of Notes” entry and initial them.
7. In order to demonstrate continuity of the project and to preclude questioning of the integrity of the data collection process, pages and SESD-generated forms should not be removed from bound logbooks under any circumstances.
8. Data or other information that has been entered incorrectly will be corrected by drawing a line through the incorrect entry and **initialing and dating** the lined-through entry. Under no circumstances should the incorrect material be erased, made illegible or obscured so that it cannot be read.
9. If pre-printed adhesive labels are used in logbooks or bound forms to facilitate organization of information entry, the field investigator who is responsible for taking notes will sign the label with the signature beginning on the label and ending on the page of the logbook such that the label cannot be removed without detection.

2.3 Logbook Entry Information

2.3.1 General Information Required in All Logbooks

The following information will be included either on the front cover or the first page of **all logbooks**:

1. Project name
2. Project location
3. Project identification number
4. Project leader (full name)
5. Sample team leader (full name) and initials
6. Sample team member(s) (full name) and initials

2.3.2 Information Required for Sample Collection

In addition to the information listed in Section 2.3.1, the following information will be included in all logbooks when **samples** are collected:

1. Applicable SESD Operating Procedures for field sampling
2. Date and time of collection
3. Station identification
4. Sample identification

5. Method of collection
6. Number and type of containers
7. Sample collection equipment
8. SESD equipment identification number, if applicable
9. Physical description of sample
10. Matrix sampled
11. Sample team member duties (calibration, collection, deployment, etc.)
12. Sample preservation (including ice), if applicable
13. Conditions that may adversely impact quality of samples, if applicable (rain, wind, smoke, dust, extreme temperature, etc.)
14. GPS coordinates (Non-logging GPS units), if applicable
15. Location of electronic data file backups, if applicable
16. Monitoring of condition of ice in coolers or sampler
17. Other pertinent information.

2.3.3 Information Required for Field Measurements

In addition to the information listed in Section 2.3.1, the following information will be included in all logbooks when **measurements** are conducted:

1. Applicable SESD Operating Procedures for field measurement
2. Date and time of measurement or deployment
3. Sample identification, if appropriate
4. Station identification
5. Sample measurement equipment
6. SESD sample measurement equipment identification number
7. Manufacturer name, lot number and expiration date of all buffers and standards*
8. Calibration information, including before and after calibration readings*
9. Meter end check information
10. Deployment depth and total depth, if applicable
11. Pinger identification number and frequency for deployed equipment, if applicable
12. Time of retrieval for deployed equipment, if applicable
13. Physical description of matrix
14. Sample team member duties (calibration, collection, deployment, etc.)
15. Measurement values for non-logging equipment
16. GPS coordinates (non-logging GPS units), if applicable
17. Location of electronic data file backups, if applicable
18. Ambient air temperature, where applicable
19. Conditions that may adversely impact quality of measurement (Ex. temperature extremes)
20. Maintenance performed, if applicable

21. Meter malfunctions, if applicable
22. Other pertinent information

* Entry of calibration information in logbooks is only required for calibrations conducted in the field. All calibrations conducted at the Field Equipment Center or SESD laboratory will be recorded in the appropriate equipment tracking logbook.

2.3.4 Additional Information for Inclusion

The following information may be included in logbooks as appropriate:

1. Maps/sketches
2. Photographic or videographic log
3. Process diagrams

Region 4
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Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

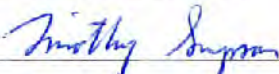
Title: Field Sampling Quality Control

Effective Date: February 5, 2013

Number: SESDPROC-011-R4

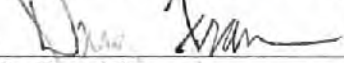
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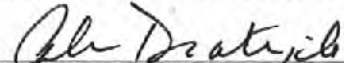
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Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the Document Control Coordinator.

History	Effective Date
<p>SESDPROC-011-R4, <i>Field Sampling Quality Control</i>, replaces SESDPROC-011-R3</p> <p>General: Corrected any typographical, grammatical and/or editorial errors.</p> <p>Cover Page: The Enforcement and Investigations Branch Chief was changed from Archie Lee to Danny France. The Ecological Assessment Branch Chief was changed from Bill Cosgrove to John Deatruck. The FQM was changed from Liza Montalvo to Bobby Lewis.</p> <p>Section 2.5: Modified the statement to read: Chemical preservatives used will be supplied by the Region 4 laboratory or purchased by the BFEM.</p> <p>Section 3.3.3: Modified section to read that SESD will generally use chemical preservatives stored in individual single-use vials. The chemical preservative will be tested prior to use for the appropriate analytes. The use of pre-tested, single-use vials eliminates the need to routinely collect preservative blanks in the field. If the preservatives are stored in containers that will be used to preserve multiple samples, blanks will be collected to evaluate the potential for cross-contamination resulting from the preservation process. Added Note for bullet 1. Modified bullet number 9 to read Water Filter Blank.</p> <p>Section 4.1: Modified first sentence to read - Records will be kept by FEC personnel. Modified bullet 1 to only apply to the FEC water system. Section 4.1, #5: - Added tubing to bullet number 5. Created bullet number 6. Moved last two sentences from bullet number 5 to create bullet number 7.</p> <p>Section 4.2: Moved testing requirements for Teflon® tubing from bullet number 1 to bullet number 6. Added requirements to bullets 2 and 4 that organic-free water for VOC samples is provided by the ASB laboratory.</p> <p>Section 4.4.1: Changed requirement for the EAB QA Officer from submitting an annual QA report to a quarterly report. Modified paragraph to read - Each branch QAO will compile a quarterly report of field quality assurance data and forward the report to the FQM.</p>	February 5, 2013
<p>SESDPROC-011-R3, <i>Field Sampling Quality Control</i>, replaces SESDPROC-011-R2</p> <p>Cover Page: The Enforcement and Investigations Branch Chief was changed from Antonio Quinones to Archie Lee. The FQM was changed from Laura Ackerman to Liza Montalvo.</p>	October 15, 2010

<p>Section 1.2: Added the following statement: Mention of trade names or commercial products does not constitute endorsement or recommendation for use.</p> <p>Section 1.3: Omitted reference to the H: drive of the LAN.</p> <p>Section 2.9: On the first paragraph, replaced the word “will” with “should” on the two instances where “will” was mentioned.</p> <p>Section 4.2, Subsection 1: Removed volatiles from list of analyses for 1-liter amber containers. Added volatile organic compound to list of analyses for 8-oz. clear glass containers.</p>	
<p>SESDPROC-011-R2, <i>Field Sampling Quality Control</i>, replaces SESDPROC-011-R1</p> <p>Revision History Changed Field Quality Manager to Document Control Coordinator.</p> <p>Section 1.3 Changed Field Quality Manager to Document Control Coordinator.</p> <p>Section 2.6 Added item #8.</p> <p>Section 4.1 Added clarifying language to item #5.</p> <p>Section 4.2 Added “volatiles” to item #1. Added “Note” at end of item #1.</p> <p>Section 4.4.1 Changed “quarter” to “year” in second paragraph.</p>	January 28, 2008
<p>SESDPROC-011-R1, <i>Field Sampling Quality Control</i>, replaces SESDPROC-011-R0.</p> <p>Section 1.3 Modified requirements for maintaining official copy of procedure.</p> <p>Section 1.4 Stated that variability should be accounted for depending on the data quality objectives for the study. Added definitions for branch field equipment manager and de-ionized water. Removed definitions for representative sample, sample representativeness, accuracy, precision, and bias.</p> <p>Section 2.5 Records of sample preservation, including ice, should be documented in the field log books.</p> <p>Section 2.6</p>	October 19, 2007

<p>Added the SESD glove and bottle return policy. The glove and bottle return policy was previously in SESDPROC-108-R0, <i>Equipment and Supply Management</i>.</p> <p>Section 3.3 Stated that variability samples will be collected in accordance to project DQOs. Removed definition for “major project”.</p> <p>Section 3.5 Added double volume requirement for SVOC/pesticide/PCB MS/MSD sample collection.</p> <p>Section 4.1 Modified requirements for servicing both field and FEC organic-free water systems from quarterly to at least once per 180 days.</p> <p>Section 4.2, Subsection 1 Added analytical requirements for testing of Teflon® tubing.</p> <p>Section 4.2, Subsection 5 Modified Silastic® or Tygon® tubing testing requirements to state that new lot are required to be tested quarterly.</p> <p>Section 4.4.2 Modified requirements that project leaders provide final report to QAO. Project Leaders will provide QA data to QAO.</p>	
<p>SESDPROC-011-R0, <i>Field Sampling Quality Control</i>, Original Issue</p>	<p>February 5, 2007</p>

TABLE OF CONTENTS

1	General Information	6
1.1	Purpose.....	6
1.2	Scope/Application	6
1.3	Documentation/Verification.....	6
1.4	Definitions.....	6
1.4.1	<i>Sample</i>	<i>6</i>
1.4.2	<i>Variability</i>	<i>6</i>
1.4.3	<i>Grab Sample</i>	<i>7</i>
1.4.4	<i>Composite Sample</i>	<i>7</i>
1.4.5	<i>De-ionized Water.....</i>	<i>8</i>
1.4.6	<i>Branch Field Equipment Manager</i>	<i>8</i>
1.5	References	8
2	Field Sampling Quality Control Considerations.....	10
2.1	Experience Requirements	10
2.2	Traceability Requirements.....	10
2.3	Chain-of-Custody	10
2.4	Sampling Equipment Construction Material.....	10
2.5	Sample Preservation	11
2.6	Sample Collection Precautions	11
2.7	Sample Handling and Mixing	12
2.8	Special Handling of Samples for Volatile Organic Compounds Analysis ..	13
3	Quality Control Samples	15
3.1	Control Sample.....	15
3.2	Background Sample.....	15
3.3	Variability Samples.....	15
3.3.1	<i>Spatial Variability Duplicate</i>	<i>15</i>
3.3.2	<i>Temporal Variability Duplicate</i>	<i>16</i>
3.3.3	<i>Sample Handling Variability</i>	<i>16</i>
3.4	Spikes	19
3.5	Matrix Spike/Matrix Spike Duplicate Samples for Water and Soil Samples for Organic Compounds Analyses.....	19
3.6	Matrix Spike/Matrix Spike Duplicate Samples for Water and Soil Samples for Inorganic Analyses.....	20
3.7	Special Quality Control Procedures for EPA Contract Laboratories	20
3.8	Special Quality Control Procedures for Dioxins and Furans	20
4	Internal Quality Control Procedures	22
4.1	Traceability Requirements.....	22
4.2	Specific Quality Control Checks.....	23
4.3	Quality Control for Special Order Equipment and Supplies	24
4.4	Quality Control Evaluation and Corrective Action.....	24
4.4.1	<i>Quality Assurance Reports</i>	<i>25</i>

Contents

1 General Information

1.1 Purpose

This document describes procedures established to ensure the quality of SESD field sampling activities, including Field Equipment Center (FEC) operations involving preparation of sampling and support equipment for field operations. Collectively, these procedures ensure that field sampling teams are provided with equipment that is suitable for sampling use, and that field sampling is conducted using proper procedures, resulting in the collection of representative samples. Strict adherence to these procedures forms the basis for an acceptable field sampling quality assurance program.

1.2 Scope/Application

The procedures contained in this document are to be used by field investigators when collecting and handling samples in the field and when preparing sampling equipment for SESD field investigations. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 Definitions

1.4.1 Sample

A part of a larger lot, usually a volume, area, period or population.

1.4.2 Variability

The range or “distribution” of results around the mean value obtained from samples within a population. There are three types of variability which should be measured or otherwise accounted for in field sampling, depending on the data quality objectives (DQO) for the study:

1. Temporal Variability

Temporal variability is the range of results due to changes in contaminant concentrations over time. An example would be the range of concentrations obtained for a given parameter in wastewater samples collected at different times from an outfall where contaminant concentrations vary over time.

2. Spatial Variability

Spatial variability is the range of results due to changes in contaminant concentrations as a function of their location. An example would be the range of concentrations obtained for a given parameter in surface soil from a site where discrete "hot spots" are present due to localized releases of contaminants on otherwise uncontaminated soil.

3. Sample Handling Variability

Sample handling variability is the range of results due to the sample collection and handling techniques used by the sampler. This variability manifests itself as a positive bias due to errors such as unclean sampling equipment, cross contamination, etc., or a negative bias due to improper containers or sample preservation.

1.4.3 Grab Sample

An individual sample collected from a single location at a specific time or period of time. Grab samples are generally authoritative in nature.

1.4.4 Composite Sample

A sample collected over a temporal or spatial range that typically consists of a series of discrete, equal samples (or "aliquots") which are combined or "composited." A composite sample represents the average characteristics of the population under consideration. Four types of composite samples are listed below:

1. Time Composite (TC) – a sample comprised of a varying number of discrete samples or "aliquots" collected at equal time intervals during the compositing period. The TC sample is typically used to sample wastewater or streams.
2. Flow Proportional Composite (FPC) – A sample consisting of discrete samples or "aliquots" collected at a rate proportional to flow. The aliquots are collected during the compositing period by either a time-varying/constant volume (TV/CV) method ("automated flow proportioning") or a time-constant/varying volume (TC/VV) method ("manual flow proportioning"). The TV/CV method is typically used with automatic

samplers that are paced by a flow meter. The TC/VV method is a manual method that individually proportions a series of discretely collected aliquots. The FPC is typically used when sampling wastewater.

3. Areal Composite – a sample composited from individual, equal aliquots collected on an areal or horizontal cross-sectional basis. Each aliquot is collected in an identical manner. Examples include sediment composites from quarter-point sampling of streams and soil samples from within grids.
4. Vertical Composite – a sample composited from individual, equal aliquots collected from a vertical cross section. Each aliquot is collected in an identical manner. Examples include vertical profiles of soil/sediment columns, lakes, and estuaries.

1.4.5 De-ionized Water

Tap water that has been treated by passing it through a standard de-ionizing resin column. At a minimum, the finished water should contain no detectable heavy metals or other inorganic compounds (i.e., at or above analytical detection limits) as defined by a standard Inductively Coupled Argon Plasma Spectrophotometer (ICP) (or equivalent) scan. De-ionized water obtained by other methods is acceptable, as long as it meets the above analytical criteria. Organic-free water may be substituted for de-ionized water.

1.4.6 Branch Field Equipment Manager

Staff, designated by management, who are responsible for ensuring that the procedures for Equipment Inventory and Management are followed. At least one Branch Field Equipment Manager (BFEM) will be designated for the Enforcement and Investigations Branch (EIB) and the Ecological Assessment Branch (EAB).

1.5 References

SESD Safety, Health and Environmental Management Program (SHEMP) Manual, Most Recent Version

SESD Operating Procedure for Sample and Evidence Management (SESDPROC-005-most recent version)

SESD Operating Procedure for Competency and Proficiency Testing, (SESDPROC-006, most recent version)

SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108-most recent version)

SESD Operating Procedure for Sediment Sampling (SESDPROC-200-most recent version)

SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205-most recent version)

SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206-most recent version)

SESD Operating Procedure for Soil Sampling (SESDPROC-300-most recent version)

SESD Operating Procedure for Waste Sampling (SESDPROC-302-most recent version)

USEPA Region 4 Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM), November 2001

USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM), Most Recent Version

Loan-In Form (SESDFORM-011, most recent version)

SESD Operating Procedure for Packing, Marking, Labeling, and Shipping of Environmental and Waste Samples (SESDPROC-209, most recent version)

SESD Operating Procedure for Training (SESDPROC-007-most recent version)

SESD Operating Procedure for Corrective Action (SESDPROC-009-most recent version)

SESD Guidance for Design and Installation of Monitoring Wells (SESDGUID-101-most recent version)

2 Field Sampling Quality Control Considerations

This section provides guidelines for establishing quality control procedures for sampling activities. Strict adherence to all of the standard operating procedures outlined in this subsection forms the basis for an acceptable sampling quality assurance program.

2.1 Experience Requirements

There is no substitute for field experience. This field experience will be gained by on-the-job training using the "buddy" system. Each new investigator will accompany an experienced employee on as many different types of field studies as possible. During this training period, the new employee will be permitted to perform all facets of field investigations, including sampling, under the direction and supervision of senior investigators. Specific requirements covering experience, competency and proficiency are found in the SESD Operating Procedure for Competency and Proficiency Testing (SESDPROC-006) and SESD Operating Procedure for Training (SESDPROC-007).

2.2 Traceability Requirements

All sample collection and measurement activities will be traceable through field records to the person collecting the sample or making the measurement. All maintenance and calibration records for sampling and measurement equipment (where appropriate) will be kept so that they are similarly traceable. The SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108) contain specific procedures to be followed that ensure traceability.

2.3 Chain-of-Custody

Specific chain-of-custody procedures are included in SESD Operating Procedure for Sample and Evidence Management (SESDPROC-005). These procedures will ensure that evidence collected during an investigation will withstand scrutiny during litigation. To assure that procedures are being followed, it is recommended that field investigators or their designees audit chain-of-custody entries, tags or labels, field notes, and any other recorded information for accuracy. Additionally, the SESD FQM will randomly conduct reviews of project files to ensure that quality procedures are being followed.

2.4 Sampling Equipment Construction Material

Sampling equipment construction materials can affect sample analytical results. Field investigators will ensure the sample equipment construction material will not introduce contaminants to the sample being collected.

2.5 Sample Preservation

Samples for some analyses must be preserved in order to maintain their integrity. Preservatives required for routine analyses of samples collected are found in the USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM). Chemical preservatives used will be supplied by the Region 4 laboratory or purchased by the BFEM. All samples requiring preservation should be preserved immediately upon collection in the field. Records of sample preservation, including ice, will be documented in the field log books.

Samples that **should not** be preserved in the field are:

1. Those collected within a hazardous waste site that are known or thought to be highly contaminated with toxic materials which may be highly reactive. Barrel, drum, closed container, spillage or other source samples from hazardous waste sites are not to be preserved with any chemical.
2. Those that have extremely low or high pH or samples that may generate potentially dangerous gases if they were preserved according to the ASBLOQAM.

All samples preserved with chemicals will be clearly identified by indication on the sample tag or label that the sample is preserved. If samples normally requiring preservation were not preserved, field records should clearly specify the reason. Samples shipped by air will not be preserved with nitric acid, hydrochloric acid, sodium hydroxide or sulfuric acid in excess of the amount specified in the ASBLOQAM.

2.6 Sample Collection Precautions

In order to prevent cross-contamination during sample collection, the following precautions will be taken:

1. A clean pair of new, non-powdered, disposable latex gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not come into contact with the media being sampled.
2. Sample containers for source samples or samples suspected of containing high concentrations of contaminants will be placed in separate plastic bags immediately after collecting, tagging, etc.
3. If possible, environmental (low concentration) samples and source or waste samples (high concentration) should be collected by different field teams. If different field teams cannot be used, all environmental samples should be collected first and placed in separate ice chests or shipping containers. Samples

of waste or highly contaminated samples should never be placed in the same ice chest as environmental samples. Ice chests or shipping containers for source or waste samples or any samples suspected to contain high concentrations of contaminants will be lined with new, clean, plastic bags.

4. If possible, one member of the field sampling team should record all of the field notes, collect GPS data, etc., while the other members collect the samples.
5. When sampling surface water and sediment at the same location, the water sample should always be collected before the sediment sample is collected.
6. Sample collection activities should proceed progressively from the least suspected contaminated area to the most suspected contaminated area.
7. Investigators should use equipment constructed of Teflon®, stainless steel, or glass that has been properly pre-cleaned according to either the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or the SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206) for collection of samples for trace metals or organic compounds analyses. Teflon® or glass is preferred for collecting samples where trace metals are of concern. Equipment constructed of plastic or PVC should not be used to collect samples for trace organic compounds analyses.
8. Field investigators should ensure the sample containers they are using have been verified as suitable for the analyses that will be conducted on the samples through the quality control procedures discussed in Section 4 of this procedure.

Upon returning from the field, un-used sample containers will be examined by project leaders to determine whether bottles should be discarded, recycled or re-shelved for use on other projects. A load-in form (SESDFORM-011) will be completed and signed by project leaders to identify the future use of sample containers returning from the field. Opened boxes of sampling containers that can be re-used, will be segregated from sealed boxes of new containers.

Opened bags of latex gloves returning from the field will be segregated from unopened gloves and will not be re-used for sample collection on other projects.

2.7 Sample Handling and Mixing

Once a sample has been collected, it may have to be transferred into separate containers for different analyses. Sample transfer should be done as soon as possible. If necessary, aqueous samples may be collected into a single, larger container for homogenization and transferred into individual sample containers. However, aqueous samples collected for volatile organic compounds, oil and grease, bacteria, sulfides and phenols analyses may not be transferred using this procedure.

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is representative of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

1. The material in the sample pan should be divided into quarters and each quarter should be mixed individually.
2. Two quarters should then be mixed to form halves.
3. The two halves should be mixed to form a homogenous matrix.

This procedure should be repeated several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction and occasionally turning the material over.

2.8 Special Handling of Samples for Volatile Organic Compounds Analysis

Water samples to be analyzed for volatile organic compounds should be stored in 40-ml septum vials with screw cap and Teflon®-silicone disk in the cap to prevent contamination of the sample by the cap. The disks should be placed in the caps (Teflon® side to be in contact with the sample) in the laboratory prior to the beginning of the field investigation.

The vials should be completely filled to prevent volatilization, and extreme caution should be exercised when filling a vial to avoid any turbulence which could also produce volatilization. The sample should be carefully poured down the side of the vial to minimize turbulence. As a rule, it is best to gently pour the last few drops into the vial so that surface tension holds the water in a convex meniscus. The cap is then applied and some overflow is lost, but the air space in the bottle is eliminated. After capping, turn the bottle over and tap it to check for bubbles. If a bubble or bubbles are present, the vial should be topped off using a minimal amount of sample to re-establish the meniscus. Care should be taken not to flush any preservative out of the vial during topping off. If, after topping off and capping the vial, bubbles are still present, a new vial should be obtained and the sample re-collected.

Soil and sediment samples for VOC analyses should be collected and handled as specified in the SESD Operating Procedure for Soil Sampling (SESDPROC-300), Waste Sampling (SESDPROC-302) or the SESD Operating Procedure for Sediment Sampling (SESDPROC-200). Soil and sediment samples collected for VOC analyses should not be mixed.

2.9 Sample Storage and Transport

After collection, sample handling should be minimized. Field investigators should use extreme care to ensure that samples are not contaminated during storage. Environmental and waste samples are typically stored in coolers. To reduce the risk of cross contamination, smaller sample containers such as 8 ounce glass jars, 40 ml VOA vials, and one-liter amber bottles should be placed inside of sealed, plastic bags before being placed in the cooler. If ice is required for preservation of the samples, the ice should be contained in a plastic bag or some equivalent container to prevent the potential for cross contamination of the samples by water produced from melting ice. If ice is used, the coolers should be checked regularly and water should be drained as needed. Custody of samples will be maintained according to the SESD Operating Procedure for Sample and Evidence Management (SESDPROC-005).

Samples will either be transported to the analytical laboratory by field investigators or shipped by common carrier. Shipping of samples will be conducted in accordance with the SESD Operating Procedure for Packing, Marking, labeling, and Shipping of Environmental and Waste Samples (SESDPROC-209).

3 Quality Control Samples

Quality control samples are collected during field studies for various purposes, among which are to isolate site effects (control samples), to define background conditions (background sample), and to evaluate field/laboratory variability (spikes and blanks, trip blanks, duplicate, split samples, etc.).

3.1 Control Sample

A control sample is typically a discrete grab sample collected to isolate a source of contamination. Isolation of a source could require the collection of both an upstream sample at a location where the medium being studied is unaffected by the site being studied, as well as a downstream control which could be affected by contaminants contributed from the site under study.

3.2 Background Sample

A background sample (usually a grab sample) is collected from an area, water body or site similar to the one being studied, but located in an area known or thought to be free from pollutants of concern.

3.3 Variability Samples

Variability may be defined as a variation in concentrations of compounds or analytes across a site or area of investigation or variations, across time, of waste streams or surface water bodies. Variation can also be introduced during sample handling. The following procedures are used to assess and evaluate variability. When appropriate, spatial duplicate grab and/or composite samples should be collected during investigations and studies in accordance to the project DQOs. In general, no more than ten percent of all samples should be collected as spatial duplicates.

3.3.1 Spatial Variability Duplicate

The following spatial duplicate sampling procedures should be used during the collection of samples as a measure of variability within the area represented by the sample. These samples should be collected at the same time, using the same procedures, the same type of equipment, and in the same types of containers as the original samples. They should also be preserved in the same manner and submitted for the same analyses as the required samples.

Spatial variability duplicate samples are typically collected during investigations where samples are collected from grids that are positioned at fixed intervals over the study area and a sample collection pattern is established within the grids. Spatial variability duplicate samples are collected using the same compositing pattern as the original sample and are collected within the same general area of representativeness, however the pattern is shifted relative to the original aliquot locations. This amount and direction of shift for the duplicate sample is

dependent upon the size of the grid or area being sampled and should be specified in the QAPP for the investigation. Data from spatial duplicates will be examined by the investigation project leader to determine if the observed spatial variability is acceptable, based on the investigation or study objectives.

3.3.2 Temporal Variability Duplicate

When appropriate, temporal variability at a given sampling location will be measured by collecting temporal duplicate samples. These samples will be collected from the same sampling location, using the same techniques and the same type of equipment, but at a time different from the original sample. The time selected for the temporal duplicate sample will be similar to the time or span of time specified for the original sample in the project work plan. Data from temporal duplicates will be examined by the project leader to determine if samples represent the time span intended in the project work plan.

3.3.3 Sample Handling Variability

The effectiveness of sample handling techniques will be measured by collecting split and blank samples.

Split Samples

Split samples will be collected by initially collecting twice as much volume as is normally collected. The material will be apportioned, after mixing, if appropriate, into two sets of containers. Both sets of containers will be submitted for analyses with one set designated as an "original sample," the other designated as a "split sample." Data from the split samples will be examined by the project leader to assess sample handling variability. On large studies (more than 20 samples collected), a minimum of 5 percent, but no more than 10 percent, of all samples will be collected as split samples unless required by site data quality objectives.

Blank Samples

The following blank samples will be prepared by the laboratory and obtained by the project leader prior to traveling to a sample site.

1. **Water Sample VOC Trip Blank** - A water sample VOC trip blank is required for every study where water samples are collected for VOC analysis. Sealed preserved (or unpreserved, if unpreserved vials were used during the investigation) 40-ml VOC vials will be transported to the field. Two sealed VOC vials will be submitted per trip blank sample. At least one trip blank sample will be submitted per sample shipment. Trip blanks will be prepared by lab personnel. Investigators should submit their request for trip blanks at least one week in advance of scheduled field investigations and inspections and never (except in emergency situations) less than two days in advance of scheduled field investigations and inspections. These samples should not be picked up earlier than the morning of departure for the scheduled

inspection/investigation. These trip blanks will be handled and treated in the same manner as the water samples collected for volatile organic compounds analysis on that particular study. These samples will be clearly identified on sample labels and Chain-of-Custody Records as trip blanks.

2. Soil/Sediment Sample VOC Trip Blank - A soil/sediment sample VOC trip blank is required for every study where soil and/or sediment samples are collected for VOC analysis. The required containers are specified the USEPA Region 4 ASBLOQAM. The request and pick up of the soil blank sample will be the same as for the water trip blank. En Core® containers will be transported to the field. These blanks will be handled and treated by field investigators in the same manner as the soil samples collected for VOC analysis on that particular study. These samples will be clearly identified on sample labels and Chain-Of-Custody Records as trip blanks. Two sealed En Core® containers will be submitted per trip blank sample. At least one set of trip blank samples will be submitted per sample shipment.

The following blanks are prepared in the field:

1. Sample Preservative Blanks - SESD will generally use chemical preservatives stored in individual single-use vials. The chemical preservative will be tested prior to use for the appropriate analytes. The use of pre-tested, single-use vials eliminates the need to routinely collect preservative blanks in the field. If the preservatives are stored in containers that will be used to preserve multiple samples, blanks will be collected to evaluate the potential for cross-contamination resulting from the preservation process. If preservative blanks are collected, sample containers will be filled with de-ionized water by SESD personnel and transported to the field and preserved and submitted for the same analyses as the other inorganic samples collected. These samples will be clearly identified as preservatives blanks on sample labels and the Chain-Of-Custody Record(s). At least one preservative blank for each type of preserved sample should be collected at the end of routine field investigations. In addition, one preservative blank will be collected for each multi-use bottle of preservative used.

Note: The deionized water will be generated from a water treatment unit provided by the SESD laboratory.

2. Equipment Rinsate Blanks - Equipment rinsate blanks will be collected whenever field decontamination of equipment to be re-used in sampling activities is performed.

When field cleaning of equipment is required during a sampling investigation, a piece of the field-cleaned equipment will be selected for collection of a rinse blank. At least one rinse blank will be collected during each week of sampling operations. After the piece of equipment has been field cleaned and prior to its being used for sample operations, it will be rinsed with organic-free water. The rinse water will be collected and submitted for analyses of all

constituents for which normal samples collected with that piece of equipment are being analyzed.

3. Organic-Free Water System Blanks - When using a portable organic-free water generating system in the field, a sample of the water generated by the system will be collected at least once during each week of operations. Based on the objectives of the study or investigation, it may be appropriate to collect a sample of the raw source water. The collected water sample will be submitted for analyses of all constituents for which normal samples are being analyzed.
4. Material Blanks - When construction materials are being used on a site in such a way as to have a potential impact on constituent concentrations in the sample, a sample of each material will be submitted for analysis.

Note: For drilling operations where materials are shipped directly to the site from the supplier, see SESD Guidance for Design and Installation of Monitoring Wells (SESDGUID-101) for material blank collection and reporting requirements.

5. Automatic Sampler Blanks - In general, cleaning procedures outlined in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206) should be adequate to ensure sample integrity. However, it is the standard practice of the Environmental Investigations Branch to submit automatic sampler blanks for analyses when automatic samplers are used to collect samples for organic compounds and metals analyses. Automatic sampler blanks for other standard analyses may be submitted in the event of a special investigation (e.g., criminal or civil).
6. Field Blank - A field blank is a sample that is prepared in the field to evaluate the potential for contamination of a sample by site contaminants from a source not associated with the sample collected (for example air-borne dust or organic vapors which could contaminate a soil sample). Organic-free water is taken to the field in sealed containers or generated on-site. The water is poured into the appropriate sample containers at pre-designated locations at the site. Field blanks should be collected in dusty environments and/or from areas where volatile organic contamination is present in the atmosphere and originating from a source other than the source being sampled.
7. Temperature Blank - A temperature blank is a container of water shipped with each cooler of samples requiring preservation by cooling to 6EC (ice). The temperature of the blank is measured at the time of sample receipt by the laboratory. No temperature blank is necessary for waste samples since waste samples do not require ice for preservation.
8. Wipe Sample Blank - A wipe sample blank is a sample of the material and solvent used for collecting wipe samples. The blank is handled, packaged and transported in the same manner as all other wipe samples with the exception that it is not exposed to actual contact with the sample medium.

9. Water Filter Blank - When filters are used for sampling a dissolved constituent, de-ionized water should be run through at least one filter from each lot and the filtered water submitted for the same analyses. When filters are used for chlorophyll sampling, the filter should be prepared using de-ionized water and submitted for the same chlorophyll analysis.

3.4 Spikes

Spike samples are used to measure bias due to sample handling or analytical procedures. Spike samples are typically used by SEDS to evaluate the performance of contract laboratories and are shipped directly to the CLP laboratory by the ESAT contractor.

3.5 Matrix Spike/Matrix Spike Duplicate Samples for Water and Soil Samples for Organic Compounds Analyses

Matrix spike and matrix spike duplicate (MS/MSD) samples will be submitted to the laboratory for volatile organic compounds, extractable organic compounds, pesticides/PCBs and/or herbicides analyses from at least one sampling location per project and laboratory used. One MS/MSD sample should be collected per 20 samples per media collected.

Additional volume will be required for the soil MS/MSD samples. Semi-volatile organic compounds, pesticides, and PCB analyses of soil/sediment samples require the collection of one additional eight ounce glass jar. For VOC soil/sediment samples, triple volume, i.e., nine En Cores® or nine 40 ml vials with syringe collected sample, is needed for the MS/MSD samples.

Additional volume will be required for the water MS/MSD samples. For routine full scan analysis, i.e., extractable organic compounds, pesticides and PCBs, four one-liter amber containers provide the required sample volume. Eight containers, therefore, should be submitted for the MS/MSD sample. For VOC water samples, a total of six 40-ml vials should be collected.

MS/MSD samples should be collected from a location expected to be relatively free from contamination, since the samples will be used for laboratory quality control purposes. The duplicate samples should be clearly identified as "Duplicate Sample for Matrix Spike" or "MS/MSD" on the Chain-Of-Custody Record, in the field logbook and on the Contract Laboratory Program (CLP) Traffic Report Form (if appropriate). This procedure will be followed for all projects where water samples are collected for the indicated analyses. For non-routine sampling events, the Region 4 SEDS laboratory should be consulted for specific sample volume and container requirements.

3.6 Matrix Spike/Matrix Spike Duplicate Samples for Water and Soil Samples for Inorganic Analyses

A matrix spike sample and a duplicate sample (MS/MSD) will be submitted to the laboratory for inorganic analyses from at least one sampling location per project and laboratory used. One matrix spike and duplicate sample should be collected per 20 samples per media collected per laboratory.

Soil/sediment and water samples collected for inorganic analyses will normally have sufficient sample volume to perform the matrix spike analyses without requiring the collection of extra sample volume. The project leader should designate a sample, typically one considered to be representative of background or relatively uncontaminated conditions, as the matrix spike sample. For water samples, the sample volume collected will normally provide adequate volume for the MS/MSD analyses.

MS/MSD samples should be collected from a location expected to be relatively free from contamination, since the samples will be used for laboratory quality control purposes. MS/MSD samples should be clearly identified as "Duplicate Sample for Matrix Spike" or "MS/MSD" on the Chain-Of-Custody Record, in the field logbook and on the Contract Laboratory Program (CLP) Traffic Report Form (if appropriate). This procedure will be followed for all projects where water samples are collected for the indicated analyses. For non-routine sampling events, the Region 4 SESD laboratory should be consulted for specific sample volume and container requirements.

3.7 Special Quality Control Procedures for EPA Contract Laboratories

On a case-by-case basis, field investigators may be required to collect split samples (or duplicate samples if appropriate) for analyses by either the Region 4 SESD laboratory or contract laboratories. The split samples are to be submitted to the Region 4 laboratory using established procedures. The contract laboratory involved will not be notified that samples were split, i.e., there should be no indication on Chain-Of-Custody Records or CLP Traffic Report Forms submitted to the contract laboratories that these samples were split with the Region 4 SESD laboratory.

3.8 Special Quality Control Procedures for Dioxins and Furans

The Region 4 laboratory does not conduct in-house analyses for dioxins and furans. Dioxin and furans analyses are conducted by contract laboratories. The Region 4 laboratory may accept environmental samples (soil, sediment, groundwater and surface water) suspected of being contaminated with polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF), as long as suspected PCDD and PCDF contamination is not due to RCRA hazardous waste classified as F020-023 and/or F026-028. If these environmental samples are not contaminated with an F020-023 and/or F026-028 waste, it may be analyzed for parameters other than dioxin and furans. Environmental samples known or suspected to be contaminated with the RCRA hazardous waste F020-023 and or F026-028 will not be accepted.

NOTE: Environmental samples suspected of being contaminated with RCRA hazardous waste classified in 40 CFR, 261.31 as F032 will be accepted. The F032 waste is defined as wastewaters (except those that have not come into contact with process contaminants), process residuals, preservative drippage, and spent formulations from wood preserving processes generated at plants that currently use or have previously used chlorophenolic formulations. The F032 listing does not include K001 bottom sediment sludge from the treatment of wastewater from wood preserving processes that use creosote and or pentachlorophenol. Prior to a sampling event, the project leaders should consult with the Analytical Support Branch Sample Control Coordinator to determine if the Region 4 laboratory can accept the samples. The Region 4 SESD laboratory should also be consulted for the current quality control procedures for dioxins and furans samples prior to a sampling investigation.

4 Internal Quality Control Procedures

The focus of this section is on Field Equipment Center (FEC) operations involving preparation of sampling and support equipment for field operations, as well as, field data generated under the specific sample collection quality control procedures discussed in Section 2. Quality control checks of these operations ensure that field sampling teams are provided with equipment that is suitable for sampling use, and that field sampling is conducted using proper procedures.

4.1 Traceability Requirements

Records, will be kept by FEC personnel documenting the dates of operations and the person performing operations for the following:

1. Organic-Free Water System Maintenance (FEC System) - Maintenance on the FEC organic-free water system will be performed at least once per 180 days.
2. Air Monitoring Safety Instrumentation Checkouts - Pre-loadout checks on safety monitoring instrumentation will be recorded each time they are performed. Discrepancies will be immediately reported to the Branch Safety Officer.
3. Self Contained Breathing Apparatus (SCBA) Checkouts - Pre-loadout checks on SCBAs will be recorded when they are performed. SCBA checkouts will be performed at least once per calendar quarter in the absence of loadout requests. Any discrepancies will be reported immediately to the Branch Safety Officer.
4. Other Equipment Maintenance - Maintenance performed on equipment other than that listed above will be accordance to the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). All required repairs will be reported to appropriate Branch Field Equipment Manager.
5. Tubing, Sampling Containers and Latex Gloves - The Enforcement and Investigations Branch Quality Assurance Officer (EIB QAO) is responsible for conducting verification sampling for tubing, sample containers, and latex gloves that are used during field investigations. Upon receipt, the tubing, containers and gloves are placed in the quarantine room at the FEC. A record is kept of the lot numbers for each shipment received. The EIB QAO will collect blank samples from tubing, containers and gloves within each lot received and will review the results to ensure the sample containers and gloves are suitable for use during field investigations. Once the supplies are deemed suitable, the EIB QAO will release the items for use.
6. Chemical preservatives commercially purchased will be tested prior to use. Each lot of chemical preservative will be tested for the appropriate analytes by either the BFEM or Branch QAO. Once released by Branch QAO, the preservatives can be used in the field.

7. Equipment - All equipment cleaned and wrapped for field use will be marked with the date on which preparation was completed. Equipment will be stored at the FEC in specified areas to minimize the risk of contamination while awaiting use.

4.2 Specific Quality Control Checks

When collecting samples during field investigations, it is necessary to take measures to prevent cross contamination to ensure the integrity of the data generated. The field branches conduct verification sampling of sample containers, gloves, sampling equipment, tubing and water utilized during field investigations as one of these measures. At least once per calendar quarter, the EIB QAO will conduct the following checks and issue a written report to the EIB Branch Chief and Field Quality Manager with the results.

1. Collect and submit for analyses samples of each new lot of containers received, Bottles from each lot will be tagged and sealed, then submitted for the following analyses:
 1. 1-liter Amber – extractable organics, pesticides, and PCBs.
 2. 8-oz. Clear Glass – metals, cyanide, extractable organics, pesticides, PCBs, and volatile organic compounds.
 3. 1-Liter Polyethylene – metals and cyanide.

NOTE: In addition to the quality control checks listed above, samples may be collected during field investigations for classical inorganic parameters such as nitrates, nitrites, sulfides, etc. Due to the detection levels generally required for these parameters, it is unlikely that cross contamination may occur in association with the sample containers and sampling equipment used during sample collection. Therefore, classical inorganic analyses are not conducted as part of the routine quality control checks. If the data quality objectives require additional quality control checks, bottles will be submitted to the laboratory for analyses.

2. Collect and submit for analyses a rinsate blank for each new lot of latex gloves received during the calendar quarter. Samples will be collected as rinse blanks using organic-free water. The rinsate will be submitted for analyses of VOCs, metals, cyanide, extractable organics, pesticides and PCBs. A new glove will be rinsed for each parameter (e.g., one glove for the VOC sample, another glove for metals, etc.) to avoid dilution of potential contaminants on the gloves. Water for the VOC samples should be provided by the ASB laboratory.

3. Collect and submit for analyses a sample of water from the FEC organic-free water system. The sample will be submitted for analyses of VOCs, metals, cyanide, extractable organics, pesticides and PCBs.
4. Collect and submit for analyses a rinsate blank of at least one piece of sampling or sample related equipment stored at the FEC. The sample will be submitted for analyses of VOCs, metals, cyanide, extractable organics, pesticides and PCBs. Water for the VOC samples should be provided by the ASB laboratory.
5. Collect and submit for analyses a rinsate blank for each new lot of Silastic® or Tygon® tubing used in peristaltic pump head. The sample will be submitted for metals and cyanide analysis.
6. Teflon® tubing – Collect and submit for analyses a rinsate blank for each new lot of Teflon® tubing received. Rinse blanks will be collected through the Teflon tubing. The sample will be submitted for metals, cyanide, extractable organics, volatile organic compounds, pesticides and PCBs. Water for the VOC samples should be provided by the ASB laboratory.

4.3 Quality Control for Special Order Equipment and Supplies

Some equipment and supplies ordered for specific projects are received in what can be considered ready to use condition. In order to ensure the integrity of these materials, an equipment rinsate blank will be collected from at least one item in each lot. The equipment and supplies will not be used until the QAO has reviewed the analytical data for the blanks and released the items.

4.4 Quality Control Evaluation and Corrective Action

All field investigation reports will contain a clearly identified section where the results for all field generated quality control (QC) samples are discussed and reported. Quality control data review includes but is not limited to detections of organic and inorganic compounds at any concentration in quality control blanks (i.e., trip blanks, equipment rinsate blanks, portable organic-free water system blanks, etc.).

All detections of organic and inorganic compounds will be immediately reported to the appropriate branch QAO. The project leader will analyze the results to determine if the source of contamination can be identified. If the source of contamination cannot be determined by the project leader, the branch QAO will conduct an additional review of the results to assess the source of contamination. If the source of contamination cannot be determined, the branch QAO will monitor all quality control results generated by the branch and assess the data for trends of contamination.

If it is determined by the project leader and the branch QAO that the contamination adversely impacts the data collected during the investigation, the project leader will report the results to their Section Chief and the FQM. The project leader, in consultation

with management, will determine whether the impacted data are usable or should be rejected. If data are rejected, the project leader and their management will determine whether samples must be recollected.

Data reported to the FQM will be analyzed to determine if the contamination is due to non-conforming work. If it is determined by the FQM, in consultation with management, that the contamination is due to non-conforming work, a corrective action is warranted and will be selected and implemented in a timely manner. If a corrective action is required, it must be implemented and reported according to the SESD Operating Procedure for Corrective Action (SESDPROC-009). If contamination is not due to non-conforming field work, then the source of contamination will be identified, if possible, and documented by the FQM. If the source of contamination cannot be determined, FQM will monitor all quality control results generated by SESD and assess the data for trends of contamination.

4.4.1 Quality Assurance Reports

It is each project leader's responsibility to ensure that a copy of the quality assurance data from each field investigation report is provided to their respective branch QAO. Each branch QAO will compile a quarterly report of field quality assurance data and forward the report to the FQM.

The FQM will prepare an annual quality assurance report based on the reports provided by the branch QAOs. This report will be distributed to all field investigators each year and will document and discuss all quality control issues or trends identified during the data review. This report will be retained by the FQM to document that QC measures have been taken, that the QC measures are appropriate, that the QC results are acceptable or, if not, that corrective actions were taken.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

**Title: Field Sampling and Measurement Procedures and
Procedure Validation**


Effective Date: August 30, 2012

Number: SESDPROC-012-R3

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Date: 8/23/12

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Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
<p>SESDPROC-012-R3, <i>Field Sampling and Measurement Procedures and Procedure Validation</i>, replaces SESDPROC-012-R2</p> <p>General: Corrected any typographical, grammatical, and/or editorial errors.</p> <p>Title Page: Changed the Author from Don Hunter to Bobby Lewis. Changed the Enforcement and Investigations Branch Chief from Archie Lee to Danny France. Changed the Ecological Assessment Branch Chief from Bill Cosgrove to John Deatruck. Changed the Field Quality Manager from Laura Ackerman to Bobby Lewis.</p> <p>Added Section 4 which describes when Competency testing should be conducted for implementing a new procedure after it has been validated and accepted.</p>	August 30, 2012
<p>SESDPROC-012-R2, <i>Field Sampling and Measurement Procedures and Procedure Validation</i>, replaces SESDPROC-012-R1</p> <p>General: Corrected typographical errors.</p> <p>Title Page: Changed Enforcement and Investigations Branch Chief from Antonio Quinones to Archie Lee.</p> <p>Revision History: Changed Field Quality Manager to Document Control Coordinator.</p> <p>Section 1.3: Omitted reference to the H: drive. Changed the Field Quality Manager to the Document Control Coordinator.</p>	December 18, 2009
<p>SESDPROC-012-R1, <i>Field Sampling and Measurement Procedures and Procedure Validation</i>, replaces SESDPROC-012-R0</p> <p>Revision History: Changed title for Antonio Quinones from Environmental Investigation Branch to Enforcement and Investigations Branch.</p>	November 1, 2007
SESDPROC-012-R0, Procedure Development, Revision and Validation, Original Issue	October 10, 2007

TABLE OF CONTENTS

1	General Information	4
1.1	Purpose.....	4
1.2	Scope/Application	4
1.3	Documentation/Verification	4
1.4	Definitions	4
1.4.1	<i>Standard Procedure</i>	4
1.4.2	<i>Non-Standard Procedure</i>	4
1.4.3	<i>SESD-Developed Procedure</i>	5
1.5	References	5
2	Field Sampling and Measurement Procedures	6
2.1	Standard Procedures.....	6
2.2	Non-Standard Procedures	6
2.3	SESD-Developed Procedures	7
3	Validation	8
3.1	General	8
3.2	Validation Team	8
3.3	Field Testing or Evaluation	8
3.4	Assessment	8
3.5	Acceptance/Rejection of Operating Procedure	8
3.6	Records	9
4	Procedure Implementation	10

1 General Information

1.1 Purpose

SESD field branches operating procedures are reviewed periodically, and where necessary, revised to ensure continuing suitability and conformance with applicable requirements and industry standards. Additionally, there may be the occasion where a new sampling or measurement procedure is developed internally or adapted from an existing standard or non-standard procedure. This document defines the different classes of procedures and describes the process for:

- The development and validation of new SESD procedures; and
- The development, review/validation and approval process to be followed when adopting or revising standard or non-standard procedures.

All SESD sampling and measurement procedures in use as of February 5, 2007 are considered standard procedures and do not require validation.

1.2 Scope/Application

This document applies to all standard, non-standard and SESD-developed sampling and measurement procedures.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 Definitions

The following definitions apply to this operating procedure.

1.4.1 Standard Procedure

This term applies to an established environmental sampling or measurement procedure used and accepted within the environmental industry.

1.4.2 Non-Standard Procedure

A non-standard procedure is an environmental sampling or measurement procedure that is not widely used or accepted within the environmental industry.

1.4.3 SESD-Developed Procedure

A new sampling or measurement procedure developed by the SESD field branches for internal use.

1.5 References

SESD Operating Procedure for Document Control (SESDPROC-001, most recent version)

2 Field Sampling and Measurement Procedures

2.1 Standard Procedures

SESD field investigators use standard procedures in the course of conducting field investigations. SESD procedures are subject to periodic review and where necessary, may be revised to ensure continuing suitability and conformance with applicable industry standards and requirements. SESD procedures undergoing periodic review that are updated with minor modifications are not subject to the validation process described in Section 3 of this procedure.

New standard procedures may be adopted for use by SESD field investigators. In addition, as state-of-the-art changes and new instruments, equipment, and procedures are developed and become available, it may be appropriate for SESD to develop and publish new operating procedures for field measurement and sampling activities.

New standard procedures adopted for use by the field branches will not be subject to an extensive validation as described in Section 3 of this procedure. New standard procedures will be evaluated to the extent necessary to ensure the result provided is equivalent to a currently used procedure or consistent results are achieved. Documentation supporting the use and acceptance of standard procedures adopted after February 5, 2007 will be maintained by the Field Quality Manager.

2.2 Non-Standard Procedures

When it is necessary to use a non-standard sampling or measurement procedure, the project leader will:

1. Inform the customer in writing (email or memo) that a non-standard procedure will be used;
2. Document that the customer is in agreement with the use of a non-standard procedure; and
3. Document in the project file the customer's request and the need for using a non-standard procedure (i.e., description of the project request and why it required the use of a non-standard procedure).

Non-standard procedures will be fully validated according to Section 3 of this procedure. If the non-standard procedure is not adopted into the scope of the field branches quality system prior to use, any results generated by the procedure must be clearly identified as being outside the scope of the quality system in the field investigation report.

If the field branches choose to include the non-standard procedure in the scope of their quality system, this will be done in accordance with the SESD Operating Procedure for Document Control (SESDPROC-001, most recent version).

2.3 SESD-Developed Procedures

When it is necessary for the field branches to develop procedures for their own use, it will be a planned activity and management will designate qualified personnel to develop the procedure. Management will also ensure adequate resources (time, equipment, funding, personnel, etc.) are available to aid in the development process. If a team of staff members is tasked with the procedure development, management will designate an individual to serve as the team leader. This individual will be responsible for facilitating the development process. The development process will consist of:

1. Review of relevant literature (manufacturers' documentation, scientific publications, etc.);
2. Development of an operating procedure; and
3. Validation of the procedure.

3 Validation

3.1 General

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled. SESD will validate 1) non-standard procedures; 2) SESD developed procedures; 3) standard procedures used outside their intended scope; and 4) amplifications and modifications of standard procedures to confirm that the procedures are fit for the intended use. Validation will be as extensive as is necessary to meet the needs of the given application.

3.2 Validation Team

SESD field branches management will designate members of the validation team and ensure adequate resources are available to complete the validation. Members may consist of subject matter experts (SMEs) and representatives from both field branches, as appropriate. Management will designate one team member as the team leader. The team leader will serve as the point of contact for the team and facilitate the validation process. The team will be responsible for developing a validation plan that should address, at a minimum, the objective of the validation, acceptance criteria, the level of testing necessary and peer review. The team will update the validation plan, as necessary, during the validation process.

3.3 Field Testing or Evaluation

The validation team will develop an appropriate field test or evaluation for the operating procedure. A member or members of the validation team will be present during the field test or evaluation to record information on the conditions, performance, and practicality of the proposed procedure.

3.4 Assessment

After completion of the field test or evaluation, the validation team will assess the results using the acceptance criteria established by the team in the validation plan.

3.5 Acceptance/Rejection of Operating Procedure

If the assessment indicates that the operating procedure meets the acceptance criteria, the validation team will finalize the operating procedure as written, or with modifications. If the acceptance criteria are not met, the team may continue the validation by returning to earlier steps in the validation process, as appropriate.

3.6 Records

The validation team will document all decisions and actions. The following records will be generated during the validation process:

1. Validation plan
2. Relevant literature
3. Draft and final operating procedures
4. Results of field tests/evaluations
5. Results of the assessment

The validation team leader will be responsible for maintaining the records during the validation process. Upon completion of the validation, the records will be maintained by the Field Quality Manager.

4 Procedure Implementation

After a standard or nonstandard procedure has been assessed and accepted for use, the subject matter expert will provide training, to the appropriate staff, on how to correctly implement the operating procedure. Competency will be documented, in accordance with SESD's Competency and Proficiency Testing Operating Procedure (SESDPROC-006, most recent version) for each staff member who will be assigned to implement the operating procedure.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: Field pH Measurement

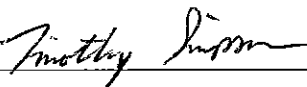
Effective Date: January 29, 2013

Number: SESDPROC-100-R3

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Date: 01/23/2013

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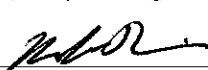
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History	Effective Date
<p>SESDPROC-100-R3, <i>Field pH Measurement</i>, replaces SESDPROC-100-R2</p> <p>General: Corrected any typographical, grammatical and/or editorial errors.</p> <p>Cover Page: The Author was changed from Ron Phelps to Timothy Simpson. The EIB Branch Chief was changed from Antonio Quinones to Danny France. The EAB Branch Chief was changed from Bill Cosgrove to John Deatrick. The Field Quality Manager was changed from Laura Ackerman to Bobby Lewis.</p> <p>Section 1.2: Added the following statement: Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.</p> <p>Section 1.3, last sentence: Added “and for maintaining records of review conducted prior to its issuance.”</p> <p>Section 1.4: Added reference to the SHEMP Manual.</p> <p>Section 1.5.1: Updated the SHEMP Manual reference to reflect that the most recent version of the Manual will be used.</p> <p>Section 2: In the first paragraph, replaced “and” in the second sentence with “or”.</p> <p>Section 3.2: Replaced the first paragraph with the following language for clarification purposes: “Many brands of instruments are commercially available for the measurement of pH incorporating a wide variety of technologies. The manufacturer’s instruction manual should be consulted for specific procedures regarding their calibration, maintenance and use. Calibration of any measurement instrument must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate. At a minimum, a two-point calibration should be conducted to ensure the accuracy of the meter. The following are basic guidelines for calibration/verification and are provided as an example:</p> <p>In Item #4, the first sentence, replaced “original buffer” to “appropriate buffer”.</p>	<p>January 29, 2013</p>

<p>Item #5 was replaced with the following: “Once the meter has been properly calibrated and verified (steps 1-4 above), it is ready for use. Rinse the probe with de-ionized water and store it according to manufacturer’s recommendations. Certain instruments may require being left on until all sample measurements are performed and the results are recorded. When collecting measurements from grab samples, certain instrument manufacturers recommend that an intermediate check(s) be performed by periodically checking the meter against a known calibration buffers if used for extended periods (> 4 hrs).</p> <p>Section 3.4: Revised the language to clarify procedures associated with operational checks.</p> <p>Item # 2 was converted to paragraphs 2.</p>	
<p>SESDPROC-100-R2, <i>Field pH Measurement</i>, replaces SESDPROC-100-R1</p> <p>Cover Page: Author was changed from Marty Allen to Ron Phelps.</p> <p>Revision History Changed Field Quality Manager to Document Control Coordinator.</p> <p>Section 1.3 Changed Field Quality Manager to Document Control Coordinator.</p> <p>Section 2 Added requirements for unattended deployment of in-situ monitoring equipment.</p> <p>Section 3.1 and 3.2, 4 Clarified requirements for routine and NPDES compliance monitoring.</p> <p>Section 3.2, 2 and 5 Added first sentence for clarification.</p>	<p>June 13, 2008</p>

<p>SESDPROC-100-R1, <i>Field pH Measurement</i>, replaces SESDPROC-100-R0</p> <p>General Deleted all references to SOSA.</p> <p>Updated referenced procedures due to changes in title names and/or to reflect most recent version.</p> <p>Replaced “shall” with “will”.</p> <p>Title Page Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove’s title from Acting Chief to Chief.</p> <p>Section 1.3 Updated information to reflect that procedure is located on the H: drive of the LAN.</p> <p>Section 1.4 Alphabetized and revised the referencing style for consistency.</p> <p>Section 2 Added last paragraph regarding stopping measurements due to environmental conditions.</p> <p>Section 3.4 Re-phrased procedure #2 for clarity.</p>	<p>November 1, 2007</p>
<p>SESDPROC-100-R0, <i>Field pH Measurement</i>, Original Issue</p>	<p>February 05, 2007</p>

TABLE OF CONTENTS

1	General Information	6
1.1	Purpose	6
1.2	Scope/Application.....	6
1.3	Documentation/Verification	6
1.4	References	6
1.5	General Precautions	7
1.5.1	<i>Safety</i>	7
1.5.2	<i>Procedural Precautions</i>	7
2	Quality Control	8
3.1	General	9
3.2	Instrument Calibration.....	9
3.3	Sample Measurement Procedures	10
3.4	Operational Check	11

1 General Information

1.1 Purpose

This document describes procedures, methods and considerations to be used and observed when conducting field pH measurements in aqueous phase environmental media, including groundwater, surface water and certain wastewaters.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when measuring the pH of aqueous phase environmental media in the field. On the occasion that SESD field personnel determine that any of the procedures described in this section cannot be used to obtain pH measurements of the media being sampled, and that another method must be used to obtain said measurements, the variant instrument and/or measurement procedure will be documented in the field logbook and subsequent investigation report, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on SESD's local area network (LAN). The Document Control Coordinator is responsible for ensuring that the most recent version of the procedure is placed on SESD's LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

United States Environmental Protection Agency (US EPA). 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

USEPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version.

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when conducting field pH measurements. Refer to the SESD Safety, Health and Environmental Management Program Procedures and Policy Manual (Most Recent Version) and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

All field pH measurements pertinent to the sampling event should be recorded in the field logbook for the event. All records, including a unique, traceable identifier for the instrument, such as a property number or serial number, should be entered according to the procedures outlined in the SESD Operating Procedure for Logbooks (SESDPROC-010) and the SESD Operating Procedure for Equipment Inventory and Management, (SESDPROC-108).

Care should be taken not to contaminate standards and samples and verify the expiration date of all standards prior to use. All meters should be calibrated, operated and maintained according to the manufacturer's specifications.

2 Quality Control

All pH meters will be maintained and operated in accordance with the manufacturer's instructions and the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). Before a meter is taken to the field, it will be properly calibrated or verified, according to Section 3.2 of this procedure, to ensure it is operating properly. These calibration and verification checks will be documented and maintained in a logbook.

The ambient temperature in the immediate vicinity of the meter should be measured and recorded in the field logbook to insure the instrument is operated within the manufacturer's specified range of operating temperatures. For instruments that are deployed for *in-situ* measurements, the temperature of the medium being monitored should be measured and recorded in the logbook prior to deployment. *In-situ monitoring equipment may be utilized in unattended deployments where autonomous logging may preclude temperature measurement prior to deployment. Because in-situ instrumentation generally has a wide range of operating temperature, the field investigator may utilize professional judgment in determining if the operating environment is suitable for unattended deployment.*

If at any time during a field investigation, it appears that the environmental conditions could jeopardize the quality of the measurement results, the measurements will be stopped. This will be documented in the field logbook.

3 Field pH Measurement Procedures

3.1 General

pH is defined as the negative logarithm of the effective hydrogen-ion concentration. For routine work, a pH meter accurate and reproducible to within 0.2 Standard Unit (S.U.) is suitable. For NPDES compliance monitoring, the pH meter should be accurate and reproducible to within 0.1 S.U. Both meters should have a range of 0 to 14 S.U.s and be equipped with a temperature-compensation adjustment.

3.2 Instrument Calibration

Many brands of instruments are commercially available for the measurement of pH incorporating a wide variety of technologies. The manufacturer's instruction manual should be consulted for specific procedures regarding their calibration, maintenance and use. Calibration of any measurement instrument must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate. At a minimum, a two-point calibration should be conducted to ensure the accuracy of the meter. The following are basic guidelines for calibration/verification and are provided as an example:

1. Verify the meter's internal temperature sensor (thermistor) against a National Institute of Standards and Technology (NIST) traceable thermometer and note any differences between the thermistor and the NIST-traceable thermometer in the logbook. If the temperatures do not agree within $\pm 4^{\circ}\text{C}$, the unit or probe must be repaired or replaced. Alternatively, if the meter can be used in a manual temperature compensation mode, the NIST-traceable thermometer may be used for temperature readings and the necessary corrections applied. Check and record the temperatures of the standards and the samples.
2. If the pH range of the sample is not known, the pH of the sample to be tested should be estimated either from historical data or by using a four-color pH indicator paper or equivalent. Using this information, calibrate the pH meter with the buffers that bracket the expected pH range. Buffer solutions are commonly pH 4, 7 and 10. It may be possible to configure the pH meter so that it can be standardized with buffers other than those in the default configuration.
3. Immerse the probe in the first buffer solution and calibrate the meter to read the correct pH. After the initial buffer calibration, calibrate the meter using other buffer solutions, as appropriate. Rinse the probe with de-ionized water and blot dry or otherwise remove excess rinse water between the different buffer solutions. Record the buffer values and temperatures used to calibrate the meter.

4. Rinse the probe with de-ionized water, blot dry or otherwise remove excess rinse water and immerse it into the appropriate buffer and read as a sample. If the meter reads within ± 0.2 S.U. of the known value of the buffer (for general applications such as ecological studies) or ± 0.1 S.U. (for regulatory applications such as NPDES or drinking Water programs), record the value indicated by the meter. If the meter is outside of the acceptable accuracy range, it should be recalibrated. If it is still outside of the acceptable accuracy range after the second calibration, the electrode and/or meter should be replaced.
5. Once the meter has been properly calibrated and verified (steps 1-4 above), it is ready for use. Rinse the probe with de-ionized water and store it according to manufacturer's recommendations. Certain instruments may require being left on until all sample measurements are performed and the results are recorded. When collecting measurements from grab samples, certain instrument manufacturers recommend that an intermediate check(s) be performed by periodically checking the meter against a known calibration buffers if used for extended periods (> 4 hrs).
6. Unless the manufacturer indicates that the meter maintains its calibration after being turned off, meters must be re-calibrated if they are turned off during their period of use.

3.3 Sample Measurement Procedures

These procedures should be followed when conducting field pH measurements of grab samples:

1. Collect a sample. If the meter's thermistor is to be used for the temperature of record for the measurement activity, the temperature should be read as soon as the reading stabilizes and prior to measuring the pH.

Note 1: When the pH meter response is slow, unstable, or non-reproducible, it may be necessary to check the conductivity. If the conductivity is lower than 20 to 30 $\mu\text{mhos/cm}$, it is permissible to add 1 ml of 1M potassium chloride solution per 100 ml of sample to improve response time for the probe. Recheck the pH and record.

Note 2: If the pH measurements are to be used for RCRA regulatory purposes and when the pH approaches the alkaline end ($\text{pH} \geq 11.0$) of the scale, the pH measurements should be made by a qualified analyst using laboratory quality equipment to control the sample at $25^{\circ}\text{C} \pm 1^{\circ}\text{C}$.

2. Immerse the probe in the sample keeping it away from the sides and

bottom of the sample container. Allow ample time for the probe to equilibrate with the sample.

3. While suspending the probe away from the sides and bottom of the sample container, record the pH.
4. Rinse the probe with de-ionized water and store it in the manufacturer's recommended storage solution until the next sample is ready.

These procedures should be followed when conducting in-situ field pH measurements:

1. Place the probe into the media to be measured and allow the pH and temperature readings to stabilize. Once the readings have stabilized, record the measurements in the logbook.
2. When deploying meters for extended periods of time, insure the measurement location is representative of average media conditions.

3.4 Operational Check

Even though it is not necessary to re-calibrate pH meters at regular intervals during the day, depending on the instrument, it may be appropriate to occasionally perform operational checks to determine if site conditions, such as an increase in temperature, have impacted the meter's performance. If an operational check is warranted, the following procedure should be followed to ensure that the performance of the meter has not changed.

1. While in use, periodically check the pH by rinsing the probe with de-ionized water, blot dry or otherwise remove excess rinse water and immerse it into the appropriate buffer solution. If the measured pH differs by ≥ 0.2 S.U. or 0.1 S.U. (depending on the application) from the buffer solution, the meter must be re-calibrated.

A post-operation instrument verification check will be performed using the appropriate buffer(s) at the end of the day or after all measurements have been taken for a particular period of operation. These measurements must be recorded in the field logbook.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: Field Specific Conductance Measurement

Effective Date: August 30, 2012

Number: SESDPROC-101-R5

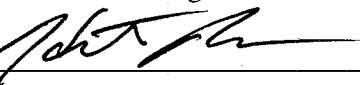
Authors

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Signature: 

Date: 08/23/2012

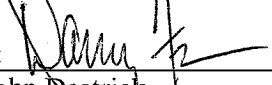
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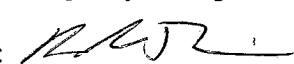
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Date: 8/23/12

Revision History

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History	Effective Date
<p>SESDPROC-101-R5, <i>Field Specific Conductance Measurement</i>, replaces SEDSPROC-101-R4</p> <p>General: Corrected any typographical, grammatical, and/or editorial errors.</p> <p>Title Page: Changed the Ecological Assessment Branch Chief from Bill Cosgrove to John Deatrick.</p> <p>Section 3.4: Added paragraphs 1 and 2 concerning performing operational checks during the day.</p>	August 30, 2012
<p>SESDPROC-101-R4, <i>Field Specific Conductance Measurement</i>, replaces SEDSPROC-101-R3</p> <p>Cover Page: Hunter Johnson was added as co-author. The EIB Branch Chief was changed from Archie Lee to Danny France. The FQM was changed from Liza Montalvo to Bobby Lewis.</p> <p>Section 3.2: Replaced the first paragraph with the following language for clarification purposes: “Many brands of instruments are commercially available for the measurement of specific conductance incorporating a wide variety of technologies. The manufacturer’s instruction manual should be consulted for specific procedures regarding their calibration, maintenance and use. Calibration of any measurement instrument must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate.”</p> <p>In the second paragraph, revised the language of the first sentence to clarify documentation procedures associated with the effect of temperature on conductivity. Replaced the second sentence with “The following are basic guidelines for calibration/verification and are provided as an example:”</p> <p>The note found in Item # 3 was revised to clarify the relationship between calibration standards and the anticipated specific conductance.</p> <p>In Item # 6, the third sentence was replaced with the following: “Certain meters may require that the instrument be left on until all sample measurements are performed and the results are recorded.” In the forth sentence, replaced “it is recommended” with “certain instrument manufacturers recommend.”</p> <p>Section 3.3: In the first sentence, replaced “must” with “should.”</p>	January 13, 2012

<p>SESDPROC-101-R3, <i>Field Specific Conductance Measurement</i>, replaces SESDPROC-101-R2</p> <p>General: Corrected any typographical, grammatical and/or editorial errors.</p> <p>Cover Page: The Author was changed from Ron Phelps to Timothy Simpson. The Enforcement and Investigations Branch Chief was changed from Antonio Quinones to Archie Lee. The FQM was changed from Laura Ackerman to Liza Montalvo.</p> <p>Section 1.2: Added the following statement: Mention of trade names or commercial products does not constitute endorsement or recommendation for use.</p> <p>Section 1.3: Omitted the reference to the H: drive of the LAN.</p> <p>Section 3.1: Added “microsiemens per centimeter ($\mu\text{S}/\text{cm}$)” in addition to micromhos per centimeter ($\mu\text{mh}/\text{cm}$) to the first sentence.</p> <p>Section 3.2: Item #3 - Added the following statement: “Fresh standards should be used for each calibration. Item #4 - Added language related to the auto-recognition of standards during meter calibration (first 3 sentences). Added items #5 and #6.</p>	<p>August 12, 2011</p>
<p>SESDPROC-101-R2, <i>Field Specific Conductance Measurement</i>, replaces SESDPROC-101-R1</p> <p>Cover Page: Author was changed from Marty Allen to Ron Phelps.</p> <p>Revision History Changed Field Quality Manager to Document Control Coordinator.</p> <p>Section 1.3 Changed Field Quality Manager to Document Control Coordinator.</p> <p>Section 2 Added requirements for unattended deployment of in-situ monitoring equipment.</p> <p>Section 3.2 Added sentence to paragraph 2 to identify minimum requirements for calibration/verification.</p> <p>Section 3.3 Moved operational check to Section 3.4.</p>	<p>June 13, 2008</p>

<p>SESDPROC-101-R1, <i>Field Specific Conductance Measurement</i>, replaces SESDPROC-101-R0</p> <p>General Deleted all references to SOSA.</p> <p>Updated referenced procedures due to changes in title names and/or to reflect most recent version.</p> <p>Title Page Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove's title from Acting Chief to Chief.</p> <p>Section 1.3 Updated information to reflect that procedure is located on the H: drive of the LAN.</p> <p>Section 1.4 Alphabetized and revised the referencing style for consistency.</p> <p>Section 2 Added last paragraph regarding stopping measurements due to environmental conditions.</p>	<p>November 1, 2007</p>
<p>SESDPROC-101-R0, <i>Field Specific Conductance Measurement</i>, Original Issue</p>	<p>February 05, 2007</p>

TABLE OF CONTENTS

1	General Information	6
1.1	Purpose.....	6
1.2	Scope/Application	6
1.3	Documentation/Verification	6
1.4	References	6
1.5	General Precautions.....	7
1.5.1	<i>Safety</i>	7
1.5.2	<i>Procedural Precautions</i>	7
2	Quality Control	8
3	Field Specific Conductance Measurement Procedures	9
3.1	General.....	9
3.2	Instrument Calibration and Verification.....	9
3.3	Sample Measurement Procedures	10
3.4	Operational Checks	11

1 General Information

1.1 Purpose

This document describes procedures, methods and considerations to be used and observed when conducting field specific conductance measurements in aqueous phase environmental media, including groundwater, surface water and certain wastewaters.

1.2 Scope/Application

The procedures contained in this document are to be used by field investigators when measuring the specific conductance of aqueous phase environmental media in the field. On the occasion that SESD field investigators determine that any of the procedures described in this section cannot be used to obtain specific conductance measurements of the media being sampled, and that another method must be used to obtain said measurements, the variant instrument and/or measurement procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

United States Environmental Protection Agency (US EPA). 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when conducting field specific conductance measurements. Refer to the SESD Safety, Health and Environmental Management Program Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

All field specific conductance measurements pertinent to the sampling event, including a unique, traceable identifier for the instrument, such as a property number or serial number, should be recorded in the field logbook for the event. All records should be entered according to the procedures outlined in the SESD Operating Procedure Logbooks (SESDPROC-010, most recent version).

Care should be taken to not contaminate standards and samples and verify the expiration date of all standards prior to use. All meters should be calibrated, operated and maintained according to the manufacturer's specifications.

2 Quality Control

All specific conductance meters will be maintained and operated in accordance with the manufacturer's instructions and the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108, most recent version). Before a meter is taken to the field, it will be properly calibrated or verified, according to Section 3.2 of this procedure, to ensure it is operating properly. These calibration and verification checks will be documented and maintained in a logbook.

The ambient temperature in the immediate vicinity of the meter should be measured and recorded in the field logbook to ensure the instrument is operated within the manufacturer's specified range of operating temperatures. For instruments that are deployed for in-situ measurements, the temperature of the medium being monitored should be measured and recorded in the logbook prior to deployment. *In-situ monitoring equipment may be utilized in unattended deployments where autonomous logging may preclude temperature measurement prior to deployment. Because in-situ instrumentation generally has a wide range of operating temperature, the field investigator may utilize professional judgment in determining if the operating environment is suitable for unattended deployment.*

If at any time during a field investigation it appears that the environmental conditions could jeopardize the quality of the measurement results, the measurements will be stopped. This will be documented in the field logbook.

3 Field Specific Conductance Measurement Procedures

3.1 General

Specific conductance is a measure of the ability of an aqueous solution to conduct an electric current and is customarily reported in microsiemens per centimeter ($\mu\text{S}/\text{cm}$) or micromhos per centimeter ($\mu\text{mhos}/\text{cm}$) at 25°C. It is important to note that if the specific conductance measurements are for NPDES reporting purposes, the meter and conductivity cell should be verified by comparing against a laboratory meter with a platinum-electrode type conductivity cell.

3.2 Instrument Calibration and Verification

Many brands of instruments are commercially available for the measurement of specific conductance incorporating a wide variety of technologies. The manufacturer's instruction manual should be consulted for specific procedures regarding their calibration, maintenance and use. Calibration of any measurement instrument must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate.

Conductivity is affected by temperature; therefore, for instruments that do not automatically compensate for temperature, the user should document temperature first so that appropriate adjustments can be made in accordance with the manufacturer's instructions and/or method. The following are basic guidelines for calibration/verification and are provided as an example:

1. Verify the meter's internal temperature sensor (thermistor) against a National Institute of Standards and Technology (NIST) traceable thermometer and note any differences between the thermistor and the NIST-traceable thermometer in the logbook. If the temperatures do not agree within $\pm 4^\circ\text{C}$, the unit must be repaired or replaced. Alternatively, if the meter can be used in a manual temperature compensation mode, the NIST-traceable thermometer may be used for temperature readings and the necessary corrections applied. Check and record the temperatures of the standards and the samples.
2. Rinse the probe with de-ionized water and blot dry before conducting the following calibration and verification checks.
3. Immerse the probe in the first standard solution and calibrate or verify the meter against that solution. Fresh standards should be used for each calibration. After the initial standard, calibrate and/or verify the meter using additional standards, as appropriate. Rinse the probe with de-ionized water and blot dry or otherwise remove excess rinse water between the different standards. Record the standard values/temperatures used to calibrate or verify the meter.

Note: Some instruments require that calibration standards reflect the

anticipated specific conductance of the media being measured.

4. Some meters will auto-recognize standards during calibration. For example, the Orion Star Series meter will auto-recognize standards 1413 $\mu\text{S}/\text{cm}$, 100 $\mu\text{S}/\text{cm}$ and 12.9 mS/cm . If the meter is calibrated in a manner where it does not auto-recognize the standard, and the meter is not accurate to within $\pm 10\%$ of the standard solution(s) known values, the meter or probe should be repaired or replaced. If this condition can be corrected by adjusting the cell constant of the probe, refer to the instruction manual and make the adjustment.
5. After calibration is complete, place the probe back into the calibration standard used and record a post-calibration reading. Record a post calibration reading for each standard used. If the meter is not accurate to within $\pm 10\%$ of the standard solution(s) known values, it should be recalibrated. If it is still outside of the acceptable accuracy range after the second calibration, the probe and/or meter should be replaced.
6. Once the meter has been properly calibrated and verified (steps 1-5 above), it is ready for use. Rinse the probe with de-ionized water and store it in the manufacturer's recommended storage solution. Certain meters may require that the instrument be left on until all sample measurements are performed and the results are recorded. When collecting measurements from grab samples, certain instrument manufacturers recommend that an intermediate check(s) be performed by periodically checking the meter against the known calibration standards if used for extended periods (> 4 hrs).

3.3 Sample Measurement Procedures

The following procedures should be followed when conducting field specific conductance measurements of grab samples:

1. Collect the sample, check and record its temperature.
2. Correct the instrument's temperature adjustment to the temperature of the sample (if required).
3. Immerse the probe in the sample keeping it away from the sides and bottom of the container. It is important that the center portion of the probe be wetted by the sample.
4. Allow meter to stabilize. Record the results in a logbook.
5. Rinse probe with de-ionized water.

The following procedures should be followed when conducting in-situ field specific conductivity measurements:

1. Place the probe into the media to be measured and allow the specific conductivity and temperature readings to stabilize. Once the readings have stabilized, record the measurements in the logbook.
2. When deploying meters for extended periods of time, ensure the measurement location is representative of average media conditions.

3.4 Operational Checks

Even though it is not necessary to re-calibrate conductivity meters at regular intervals during the day, depending on the instrument, it may be appropriate to occasionally perform operational checks to determine if site conditions, such as an extreme temperature change, have impacted the meter's performance. If an operational check is warranted, the following procedures should be followed to ensure that the performance of the meter has not changed.

Check the conductivity meter with fresh conductivity standard. Rinse the conductivity probe with deionized water, blot dry or otherwise remove excess rinse water and immerse it into the appropriate conductivity standard. If the measured conductivity value is not within $\pm 10\%$ of the standard, the probe should be re-calibrated. If the probe is still not within $\pm 10\%$ of the standard, the probe should be repaired or replaced. These measurements must be recorded in the field logbook.

A post-operation instrument verification check should be performed using the appropriate standard(s) at the end of the day or after all measurements have been taken for a particular period of operation. These measurements must be recorded in the field logbook.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

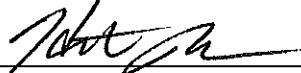
Title: Field Temperature Measurement

Effective Date: February 4, 2011

Number: SESDPROC-102-R3

Authors

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Approvals

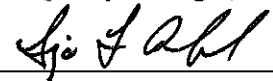
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Signature:  **Date:** 2/4/2011

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Signature:  **Date:** 2/4/11

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Signature:  **Date:** 2/4/11

Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
<p>SESDPROC-102-R3, <i>Field Temperature Measurement</i>, replaces SESDPROC-102-R2</p> <p>Cover Page: Author was changed from Ron Phelps to Hunter Johnson. The Enforcement and Investigations Branch Chief was changed from Antonio Quinones to Archie Lee. The FQM was changed from Laura Ackerman to Liza Montalvo.</p> <p>Section 1.2: Added the following statement: Mention of trade names or commercial products does not constitute endorsement or recommendation for use.</p> <p>Section 1.3: Omitted the reference to the H: drive of the LAN.</p> <p>Section 1.5: In Section 1.5.1, added “The field investigators will” to the first sentence. Deleted Section 1.5.2.</p> <p>Section 2: In the first paragraph, kept the first sentence but replaced the rest of the paragraph with the following language for clarification purposes: “Temperature measurement devices such as pH, conductivity and dissolved oxygen (DO) meter thermistors will be verified against a National Institute of Standards and Technology (NIST)-traceable thermometer before each use as described in Section 3.2. Data Sonde thermistors will be verified on a monthly basis to ensure accuracy. These verification checks are documented in the instrument’s tracking logbook.”</p> <p>Deleted the second paragraph.</p> <p>Section 3.1: In the first paragraph, replaced the first sentence with: “Field temperature measurements may be made with a field thermometer, equipment thermistor, or NIST-traceable thermometer.” In the second sentence, replaced “thermometer” with “temperature measurement device.”</p> <p>Section 3.2: Created new sections 3.2.1 and 3.2.2 as follows: Converted the “Note” to new section 3.2.1. Replaced the first sentence with “Temperature measurement devices such as field thermometers and equipment thermistors will be verified against a NIST-traceable thermometer prior to use and should agree within $\pm 4.0^{\circ}\text{C}$.”</p> <p>Converted the first paragraph to section 3.2.2. Revised the original language to clarify the verification requirements of the NIST-traceable thermometers that are used to verify other temperature measuring devices, such as field thermometers and equipment thermistors.</p>	<p>February 4, 2011</p>

<p>Section 3.3: Replaced “thermometer” with “temperature measurement device.”</p> <p>Section 3.4: Added “if not in-situ” to number 4.</p>	
<p>SESDPROC-102-R2, <i>Field Temperature Measurement</i>, Replaces SESDPROC-102-R1</p> <p>Cover Page: Author was changed from Marty Allen to Ron Phelps.</p> <p>Revision History Changed Field Quality Manager to Document Control Coordinator.</p> <p>Section 1.3 Changed Field Quality Manager to Document Control Coordinator.</p> <p>Section 2 Deleted last sentence of paragraph 1. Deleted Fisher brand stem type in paragraph 2.</p> <p>Section 3.1 Deleted “Normally” on the first sentence. Deleted the last sentence.</p> <p>Section 3.2 Changed “Calibration” to “Verification.” Added recalibration requirements.</p>	June 13, 2008
<p>SESDPROC-102-R1, <i>Field Temperature Measurement</i>, Replaces SESDPROC-102-R0</p> <p>General Updated referenced procedures due to changes in title names and/or to reflect most recent version.</p> <p>Title Page Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove’s title from Acting Chief to Chief.</p> <p>Section 1.3 Updated information to reflect that procedure is located on the H: drive of the LAN.</p> <p>Section 1.4 Alphabetized and revised the referencing style for consistency.</p> <p>Section 2 Added last paragraph regarding stopping measurements due to environmental conditions.</p>	November 1, 2007
<p>SESDPROC-102-R0, <i>Field Temperature Measurement</i>, Original Issue</p>	February 05, 2007

TABLE OF CONTENTS

1	General Information	5
1.1	Purpose	5
1.2	Scope/Application	5
1.3	Documentation/Verification	5
1.4	References	5
1.5	General Precautions	6
	1.5.1 Safety	6
2	Quality Control	7
3	Field Temperature Measurement Procedures	8
3.1	General	8
3.2	Instrument Verification	8
	3.2.1 Field thermometers and thermistors	8
	3.2.2 NIST-traceable thermometer	8
3.3	Inspections.....	8
3.4	Sample measurement procedures for thermometers/thermistors	8
3.5	Units.....	9

1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when measuring the temperature of aqueous phase environmental media, including groundwater, surface water and certain wastewaters.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when measuring the temperature of aqueous phase environmental media in the field. On the occasion that SESD field personnel determine that any of the procedures described in this section cannot be used to obtain temperature measurements of the media being sampled, and that another method or measurement instrument must be used to obtain said measurements, the variant instrument and measurement procedure will be documented in the field log book and subsequent investigation report, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

United States Environmental Protection Agency (US EPA). 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when conducting temperature measurements in the field. Refer to the SESD Safety, Health and Environmental Management Program Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. The field investigators will address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

2 **Quality Control**

All thermometers should be maintained and operated in accordance with the manufacturer's instructions and the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). Temperature measurement devices such as pH, conductivity and dissolved oxygen (DO) meter thermistors will be verified against a National Institute of Standards and Technology (NIST)-traceable thermometer before each use as described in Section 3.2. Data Sonde thermistors will be verified on a monthly basis to ensure accuracy. These verification checks are documented in the instrument's tracking logbook.

If at any time during a field investigation, it appears that the environmental conditions could jeopardize the quality of the measurement results, the measurements will be stopped. This will be documented in the field logbook.

3 Field Temperature Measurement Procedures

3.1 General

Field temperature measurements may be made with a field thermometer, equipment thermistor, or NIST-traceable thermometer. At a minimum, the temperature measurement device should be capable of measuring in 0.1°C increments.

3.2 Instrument Verification

3.2.1 Field thermometers and thermistors

Temperature measurement devices such as field thermometers and equipment thermistors will be verified against a NIST-traceable thermometer prior to use and should agree within $\pm 4.0^\circ\text{C}$. Corrections may be applied for measurements up to $\pm 4.0^\circ\text{C}$ depending on investigation objectives, but the instrument must be repaired or replaced beyond that range.

3.2.2 NIST-traceable thermometer

Verification of the NIST-traceable thermometers that are used to verify temperature measuring devices is accomplished by comparing temperature readings from the NIST-traceable thermometer to a thermometer that has an independent certification of accuracy traceable to the National Institute of Standards and Testing. Current certified thermometers are maintained by the SESD Analytical Support Branch and are called reference thermometers.

Each NIST-traceable thermometer is verified by comparing at least annually against a reference thermometer. If corrections need to be applied, they will be noted in the NIST-traceable thermometer. Depending on investigation objectives, project leaders may decide to apply the correction factor as necessary.

3.3 Inspections

All temperature measurement devices should be inspected for leaks, cracks, and/or function prior to each use.

3.4 Sample measurement procedures for thermometers/thermistors

(Make measurements in-situ when possible)

1. Clean the probe end with de-ionized water and immerse into sample.
2. If not measuring in-situ, swirl the instrument in the sample for mixing and equilibration.

3. Allow the instrument to equilibrate with the sample for at least one minute.
4. Suspend the instrument away from the sides and bottom, if not in-situ, to observe the temperature reading.
5. Record the reading in the log book. For most applications, report temperature readings to the nearest 0.5°C or to the nearest 0.1°C depending on need.

Note: Always clean the thermometer with de-ionized water or a detergent solution, if appropriate, prior to storage and/or use.

3.5 Units

Degrees Celsius (°C) or Degrees Fahrenheit (°F)

Conversion Formulas:

$$^{\circ}\text{F} = (9/5 ^{\circ}\text{C}) + 32 \quad \text{or} \quad ^{\circ}\text{C} = 5/9 (^{\circ}\text{F} - 32)$$

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Athens, Georgia

OPERATING PROCEDURE

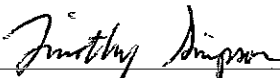
Title: Field Turbidity Measurement

Effective Date: January 29, 2013

Number: SESDPROC-103-R3

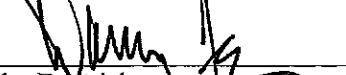
Authors

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Signature:  **Date:** 01/23/2013

Approvals

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History	Effective Date
<p>SESDPROC-103-R3, <i>Field Turbidity Measurement</i>, replaces SESDPROC-103-R2</p> <p>General: Corrected any typographical, grammatical and/or editorial errors.</p> <p>Cover Page: The Author was changed from Ron Phelps to Timothy Simpson. The Enforcement and Investigations Branch Chief was changed from Antonio Quinones to Danny France. The FQM was changed from Laura Ackerman to Bobby Lewis.</p> <p>Section 1.2: Added the following statement: Mention of trade names or commercial products does not constitute endorsement or recommendation for use.</p> <p>Section 1.3: Omitted the reference to the H: drive of the LAN.</p> <p>Section 1.5.1: Updated the SHEMP Manual reference to reflect that the most recent version of the Manual will be used.</p> <p>Section 2: In the first paragraph, replaced “and” in the second sentence with “or”.</p> <p>Section 3.2: Replaced “Meter” with “Instrument” in section title.</p> <p>Replaced the first paragraph with the following language: “Many brands of instruments are commercially available for the measurement of turbidity incorporating a wide variety of technologies. The manufacturer’s instruction manual should be consulted for specific procedures regarding their calibration, maintenance and use. Calibration of any measurement instrument must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate. Depending on the instrument, the verification and calibration can differ slightly. If the instrument readings do not agree within $\pm 10\%$ of the calibration standards, the unit must be recalibrated, repaired or replaced. The following are basic guidelines for calibration/verification of meters and are provided as an example:”</p> <p>Removed section on calibration and verification of the HACH 2100P Turbidimeter.</p> <p>Added Section 3.2.1, Meter Calibration and Verification, that includes information on the calibration and verification of the 2100Q Turbidimeter.</p> <p>Replaced Section 3.3, Probe Calibration and Verification, with Section 3.2.1, Probe Calibration and Verification.</p>	<p>January 29, 2013</p>

<p>Section 3.3: Added “Depending on the meter, the sample measurement procedure can differ slightly.”</p> <p>Converted Section 3.3 into Section 3.3.1, Grab Sample Measurement, and Section 3.3.2, <i>In-Situ</i> Measurement.</p> <p>Section 3.3.1: In Item #3 replaced “Press I/O and the instrument will turn on.” with “Turn instrument on.”</p> <p>In Item #5 and #6 added “If appropriate”.</p> <p>In Item #8 added “or rinse out with sample water prior to the next reading”.</p> <p>Section 3.5: Replaced the section with the following language: “Even though it is not necessary to re-calibrate turbidity meters at regular intervals during the day, depending on the instrument, it may be appropriate to occasionally perform operational checks to determine if site conditions, such as an increase in temperature, have impacted the meter’s performance. If an operational check is warranted, the following procedure should be followed to ensure that the performance of the meter has not changed.</p> <p>While in use, periodically check the turbidity by rinsing the probe with de-ionized water, blot dry or otherwise remove excess rinse water and immerse it into the appropriate calibration standard. If the measured turbidity differs by $\pm 10\%$ (depending on the application) from the calibration standard, the meter must be re-calibrated.</p> <p>A post-operation instrument verification check will be performed using the appropriate standard(s) at the end of the day or after all measurements have been taken for a particular period of operation. These measurements must be recorded in the field logbook.”</p>	
<p>SESDPROC-103-R2, <i>Field Turbidity Measurement</i>, replaces SESDPROC-103-R1</p> <p>Cover Page: Author was changed from Marty Allen to Ron Phelps.</p> <p>Revision History: Changed Field Quality Manager to Document Control Coordinator.</p> <p>Section 1.3: Changed Field Quality Manager to Document Control Coordinator.</p>	<p>June 13, 2008</p>

<p>SESDPROC-103-R1, <i>Field Turbidity Measurement</i>, replaces SESDPROC-103-R0</p> <p>General Deleted all references to SOSA.</p> <p>Updated referenced procedures due to changes in title names and/or to reflect most recent version.</p> <p>Title Page Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove's title from Acting Chief to Chief.</p> <p>Section 1.3 Updated information to reflect that procedure is located on the H: drive of the LAN.</p> <p>Section 1.4 Alphabetized and revised the referencing style for consistency.</p> <p>Section 2 Added last paragraph regarding stopping measurements due to environmental conditions.</p> <p>Section 3.5 Re-phrased operational check 2 for clarity.</p>	<p>November 1, 2007</p>
<p>SESDPROC-103-R0, <i>Field Turbidity Measurement</i>, Original Issue</p>	<p>February 05, 2007</p>

TABLE OF CONTENTS

1	General Information	6
1.1	Purpose	6
1.2	Scope/Application	6
1.3	Documentation/Verification	6
1.4	References	6
1.5	General Precautions	7
1.5.1	<i>Safety</i>	7
1.5.2	<i>Procedural Precautions</i>	7
2	Quality Control	8
3	Field Turbidity Measurement Procedures	9
3.1	General	9
3.2	Instrument Calibration and Verification	9
3.2.1	<i>Meter Calibration and Verification</i>	9
3.2.2	<i>Probe Calibration and Verification</i>	10
3.3	Sample Measurement Procedures	11
3.3.1	<i>Grab Sample Measurement</i>	11
3.3.2	<i>In-Situ Measurement</i>	11
3.5	Operational check	12
3.6	Units	12

1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when conducting field turbidity measurements in aqueous phase environmental media, including groundwater, surface water and certain wastewaters.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when measuring turbidity of various, aqueous phase environmental media in the field. On the occasion that SESD field personnel determine that any of the procedures described in this section cannot be used to obtain turbidity measurements of the media being sampled, and that another method or turbidity measurement instrument must be used to obtain said measurements, the variant instrument and measurement procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

United States Environmental Protection Agency (US EPA). 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when conducting field turbidity measurements. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Manual (Most Recent Version) and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. When using this procedure, minimize exposure to potential health hazards through the use of protective clothing, eye wear and gloves. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

All field turbidity measurements pertinent to the sampling event should be recorded in the field logbook for the event. All records should be entered according to the procedures outlined in the SESD Operating Procedure for Logbooks (SESDPROC-010).

2 Quality Control

All turbidity meters and probes shall be maintained and operated in accordance with the manufacturer's instructions and the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). Before a meter or probe is taken to the field, it shall be properly calibrated or verified, according to Sections 3.2 and 3.3 of this procedure, to ensure it is operating properly. These calibration and verification checks shall be documented and maintained in a logbook.

The ambient temperature in the immediate vicinity of the meter should be measured and recorded in the field logbook to insure the instrument is operated within the manufacturer's specified range of operating temperatures. For instruments that are deployed for in-situ measurements, the temperature of the medium being monitored should be measured and recorded in the logbook prior to deployment. *In-situ monitoring equipment may be utilized in unattended deployments where autonomous logging may preclude temperature measurement prior to deployment. Because in situ instrumentation generally has a wide range of operating temperature, the field investigator may utilize professional judgment in determining if the operating environment is suitable for unattended deployment.*

If at any time during a field investigation, it appears that the environmental conditions could jeopardize the quality of the measurement results, the measurements will be stopped. This will be documented in the field logbook.

3 Field Turbidity Measurement Procedures

3.1 General

Turbidity is caused by suspended and colloidal matter such as clay, silt, organic and inorganic matter and microscopic organisms. Many methods are available for the measurement of turbidity including turbidimeters and optical probes. Turbidity is measured by determining the amount of scatter when a light is passed through a sample.

3.2 Instrument Calibration and Verification

Many brands of instruments are commercially available for the measurement of turbidity incorporating a wide variety of technologies. The manufacturer's instruction manual should be consulted for specific procedures regarding their calibration, maintenance and use. Calibration of any measurement instrument must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate. Depending on the instrument, the verification and calibration can differ slightly. If the instrument readings do not agree within $\pm 10\%$ of the calibration standards, the unit must be recalibrated, repaired or replaced. The following are basic guidelines for calibration/verification of meters and are provided as an example:

3.2.1 Meter Calibration and Verification

HACH 2100Q Turbidimeter:

Portable turbidimeters are calibrated with Formazin Primary Standards. The manufacturer recommends calibration with a primary standard such as StablCal® Stabilized Standards or with formazin standards every three months.

Generally only a calibration verification measurement is required in the field; however, if a calibration is needed, record a post calibration reading for each calibration standard used.

Meter Verification:

1. Push **Verify Cal** to enter the Verify menu.
2. Gently invert the liquid standard several times prior to insertion into meter. Insert the 10.0 NTU (or other defined value) Verification Standard and close the Lid.
3. Push **Read**. The display shows "Stabilizing" and then shows the result and tolerance range.

4. Push **Done** to return to the reading display. Repeat the calibration verification if the verification failed. If a meter is unable to pass verification, then that meter will need to be calibrated.

Meter Calibration:

1. Push the **CALIBRATION** key to enter the Calibration mode. Follow the instructions on the display. **Note:** Gently invert each standard several times before inserting the standard and use a non-abrasive, lint-free paper or cloth to wipe off the standards.
2. Insert the 20 NTU StablCal Standard and close the lid. Push **Read**. The display shows “Stabilizing” and then shows the result. Record the result.
3. Repeat Step 2 with the 100 NTU and 800 NTU StablCal Standard. Record both results.
4. Push **Done** to review the calibration details.
5. Push **Store** to save the results. After a calibration is complete, the meter automatically goes into the Verify Cal mode.

3.2.2 Probe Calibration and Verification

The manufacturer’s instruction manual should be consulted for specific procedures regarding probe’s calibration, maintenance and use. Their calibration must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate. The following are basic guidelines for calibration/verification of probes and are provided as an example:

1. Turn the meter “ON” and allow it to stabilize
2. Immerse the probe in the first standard solution and calibrate the probe against the solution.
3. Rinse the probe with de-ionized water, remove excess rinse water and calibrate the probe using additional standards as appropriate.
4. Record the standard values used to calibrate the meter.

3.3 Sample Measurement Procedures

Depending on the meter, the sample measurement procedure can differ slightly.

3.3.1 Grab Sample Measurement

These procedures should be followed when conducting turbidity measurements of grab samples:

1. Collect a representative sample and pour off enough to fill the cell to the fill line (about 15 mL) and replace the cap on the cell.
2. Wipe off excess water and any streaks with a soft, lint-free cloth (lens paper).
3. Turn instrument on. Place the meter on a flat, sturdy surface. Do not hold the instrument while making measurements.
4. Insert the sample cell in the instrument so the diamond or orientation mark aligns with the raised orientation mark in the front of the cell compartment. Close the lid.
5. If appropriate, select manual or automatic range selection by pressing the range key.
6. If appropriate, select signal averaging mode by pressing the Signal Average key. Use signal average mode if the sample causes a noisy signal (display changes constantly).
7. Press Read. The display will show ---- NTU. Then the turbidity is displayed in NTU. Record the result after the lamp symbol turns off.
8. Rinse the cell with de-ionized water or rinse out with sample water prior to the next reading.

3.3.2 In-Situ Measurement

These procedures should be followed when conducting in-situ turbidity measurements:

1. Place the probe into the media to be measured and allow the turbidity reading to stabilize. Once the reading has stabilized, record the measurement in the logbook.

2. When deploying meters for extended periods of time, ensure the measurement location is representative of average media conditions.

3.5 Operational check

Even though it is not necessary to re-calibrate turbidity meters at regular intervals during the day, depending on the instrument, it may be appropriate to occasionally perform operational checks to determine if site conditions, such as an increase in temperature, have impacted the meter's performance. If an operational check is warranted, the following procedure should be followed to ensure that the performance of the meter has not changed.

While in use, periodically check the turbidity by rinsing the probe with de-ionized water, blot dry or otherwise remove excess rinse water and immerse it into the appropriate calibration standard. If the measured turbidity differs by $\pm 10\%$ (depending on the application) from the calibration standard, the meter must be re-calibrated.

A post-operation instrument verification check will be performed using the appropriate standard(s) at the end of the day or after all measurements have been taken for a particular period of operation. These measurements must be recorded in the field logbook.

3.6 Units

Turbidity measurements are reported in nephelometric turbidity units (NTUs). It is important to note that if the turbidity measurements are for NPDES reporting purposes, all values above 40 NTU must be diluted with turbidity free-water and calculated by multiplying by a dilution factor.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: Groundwater Level and Well Depth Measurement

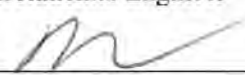
Effective Date: January 29, 2013

Number: SESDPROC-105-R2

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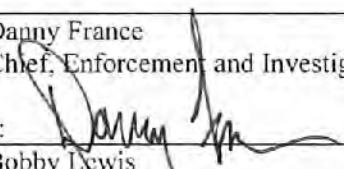
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1-22-13

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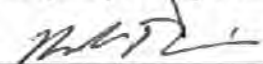
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1/22/13

Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
<p>SESDPROC-105-R2, <i>Groundwater Level and Well Depth Measurement</i>, replaces SESDPROC-105-R1</p> <p>General: Corrected any typographical, grammatical, and/or editorial errors.</p> <p>Title Page: Changed the Author from Don Hunter to Brian Striggow Changed the EIB Chief from Antonio Quinones to Danny France. Changed the FQM from Laura Ackerman to Bobby Lewis.</p> <p>Revision History: In the 3rd sentence, changed Field Quality Manager to Document Control Coordinator.</p> <p>Section 1.2: Added the following statement: Mention of trade names or commercial products does not constitute endorsement or recommendation for use.</p> <p>Section 1.3: Omitted reference to the H: drive. Changed Field Quality Manager to Document Control Coordinator.</p> <p>Section 1.4: Updated references.</p> <p>Section 2, 1st bullet: Replaced references to “calibration” of groundwater level measuring devices to “verification” of such devices. In the 1st sentence, updated the language to reflect that devices used to measure groundwater levels will be verified annually against a NIST-traceable measuring tape instead of an Invar® steel surveyor’s chain. In the 2nd sentence, added “...with an allowable error of 0.03 feet in the first 30 feet.” Added the last sentence (reference to SESDFORM-043, <i>Well Sounder Function Check and Verification</i> form).</p> <p>Section 3.1, 1st paragraph, last sentence: Rewrote the sentence for clarity. Added the 4th paragraph regarding equilibration of water levels.</p> <p>Added new Section 3.3, <i>Special Considerations for Water Level Measurements at Low Groundwater Gradient</i>. Re-numbered following sub-sections.</p> <p>Section 3.4: Added the 5th sentence and the last sentence.</p>	<p>January 29, 2013</p>

<p>SESDPROC-105-R1, <i>Groundwater Level and Well Depth Measurement</i>, replaces SESDPROC-105-R0</p> <p>General Updated referenced procedures due to changes in title names and/or to reflect most recent version.</p> <p>Title Page Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch.</p> <p>Section 1.3 Updated information to reflect that procedure is located on the H: drive of the LAN.</p> <p>Section 1.4 Alphabetized and revised the referencing style for consistency.</p>	<p>November 1, 2007</p>
<p>SESDPROC-105-R0, Groundwater Level and Well Depth Measurement, Original Issue</p>	<p>February 05, 2007</p>

TABLE OF CONTENTS

1	General Information	5
1.1	Purpose	5
1.2	Scope/Application	5
1.3	Documentation/Verification	5
1.4	References	5
1.5	General Precautions	5
1.5.1	<i>Safety</i>	5
1.5.2	<i>Procedural Precautions</i>	6
2	Quality Control Issues	7
3	Water Level and Depth Measurement Procedures	8
3.1	General	8
3.2	Specific Groundwater Level Measurement Techniques	8
3.3	Special Considerations for Water Level Measurements at Sites with Shallow Groundwater Gradient	9
3.4	Total Well Depth Measurement Techniques	9
3.5	Equipment Available	9

1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when determining water levels and depths of wells.

1.2 Scope/Application

The procedures contained in this document are to be used by field investigators to measure water levels and depths of wells. On the occasion that SESD field investigators determine that any of the procedures described in this section are either inappropriate, inadequate or impractical and that another procedure must be used for water level or depth determination, the variant procedure(s) will be documented in the field logbook and the subsequent investigation report, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network. The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when measuring water levels in wells and determining their depths. Refer to the SESD Safety, Health and

Environmental Management Program Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when measuring water levels and depths of wells:

- Special care must be taken to minimize the risk of cross-contamination between wells when conducting water level and depth measurements. This is accomplished primarily by decontaminating the sounders or other measuring devices between wells, according to SESD Operating Procedure for Field Equipment Cleaning and Decontamination, (SESDPROC-205) and maintaining the sounders in clean environment while in transit between wells.
- Water levels and well depths measured according to these procedures should be recorded in a bound logbook dedicated to the project as per SESD Operating Procedure for Logbooks (SESDPROC-010). Serial numbers, property numbers or other unique identification for the water level indicator or sounder must also be recorded.

2 Quality Control Issues

There are several specific quality control issues pertinent to conducting water level and depth measurements at wells. These are:

- Devices used to measure groundwater levels will be verified annually against a National Institute of Standards and Technology (NIST) traceable measuring tape.. These devices should check to within 0.01 feet per 10 feet of length with an allowable error of 0.03 feet in the first 30 feet. Before each use, these devices should be prepared according to the manufacturer's instructions (if appropriate) and checked for obvious damage. All verification and maintenance data should be documented electronically or recorded in a logbook maintained at the Field Equipment Center (FEC) as per the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). The functional check and tape length verification should be performed according to the instructions included in SESDFORM-043, *Well Sounder Function Check and Verification*, which also includes the form for recording the required information.
- These devices should be decontaminated according to the procedures specified in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) prior to use at the next well.

3 Water Level and Depth Measurement Procedures

3.1 General

The measurement of the groundwater level in a well is frequently conducted in conjunction with ground water sampling to determine the “free” water surface. This potentiometric surface measurement can be used to establish ground water direction and gradients. Groundwater level and well depth measurements are needed to determine the volume of water or drawdown in the well casing for proper purging.

All groundwater level and well depth measurements should be made relative to an established reference point on the well casing and should be documented in the field records. This reference point is usually identified by the well installer using a permanent marker for PVC wells, or by notching the top of casing with a chisel for stainless steel wells. By convention, this marking is usually placed on the north side of the top of casing. If no mark is apparent, the person performing the measurements should take both water level and depth measurements from the north side of the top of casing and note this procedure in the field logbook.

To be useful for establishing groundwater gradient, the reference point should be tied in with the NGVD (National Geodetic Vertical Datum) or a local datum. For an isolated group of wells, it is acceptable to use an arbitrary datum common to all wells in that group, if necessary.

Water levels should be allowed to equilibrate prior to measurement after removing sealing caps. There are no set guidelines and appropriate equilibration times can range from minutes to hours depending on well recharge, local geology and topography, and project objectives.

3.2 Specific Groundwater Level Measurement Techniques

Measuring the depth to the free ground water surface can be accomplished by the following methods. Method accuracies are noted for each of the specific methods described below.

- **Electronic Water Level Indicators** – These types of instruments consist of a spool of dual conductor wire, a probe attached to the end and an indicator. When the probe comes in contact with the water, the circuit is closed and a meter light and/or audible buzzer attached to the spool will signal contact. Penlight or 9-volt batteries are normally used as a power source. Measurements should be made and recorded to the nearest 0.01 foot.
- **Other Methods** – There are other types of water level indicators and recorders available on the market, such as weighted steel tape, chalked tape, sliding float method, air line pressure method and automatic recording methods. These methods are primarily used for closed systems or permanent monitoring wells.

Acoustic water level indicators are also available which measure water levels based on the measured return of an emitted acoustical impulse. Accuracies for these methods vary and should be evaluated before selection. Any method not capable of providing measurements to within 0.1 foot should not be used.

3.3 Special Considerations for Water Level Measurements at Sites with Shallow Groundwater Gradient

Groundwater gradients at some sites can be very shallow and if gradient and groundwater flow pattern (gradient direction) determination are part of the project objectives, it is critical that groundwater level measurements obtained from wells are as accurate as possible. Special care should be taken to allow the water level to equilibrate after removing sealing caps and the same sounder should be used for all measurements, if possible. The sounding activity should be coordinated to allow all wells to be sounded within the minimum possible time. This is particularly important in areas with potential tidal influences.

3.4 Total Well Depth Measurement Techniques

The well sounder, weighted tape or electronic water level indicators can be used to determine the total well depth. This is accomplished by lowering the tape or cable until the weighted end is felt resting on the bottom of the well. Because of tape buoyancy and weight effects encountered in deep wells with long water columns, it may be difficult to determine when the tape end is touching the bottom of the well and sediment in the bottom of the well can also make it difficult to determine total depth. Care must be taken in these situations to ensure accurate measurements. The operator may find it easier to allow the weight to touch bottom and then detect the 'tug' on the tape while lifting the weight off the well bottom. All total depth measurements must be made and recorded to the nearest 0.1 foot. As a cautionary note, when measuring well depths with the electronic water level indicators, the person performing the measurement must measure and add the length of the probe beneath the circuit closing electrodes to the depth measured to obtain the true depth. This is necessary because the tape distance markings are referenced to the electrodes, rather than the end of the probe. For electronic sounders maintained at the SESD FEC, the sounder reel will be marked with the appropriate additional length identified as the 'TD adder'.

3.5 Equipment Available

The following equipment is available for ground water level and total depth measurements:

- Weighted steel measuring tapes
- Electronic water level indicators

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: Field Measurement of Dissolved Oxygen

Effective Date: January 8, 2014

Number: SESDPROC-106-R3

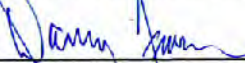
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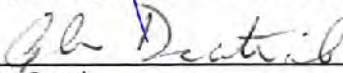
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Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the SESD Document Control Coordinator on the SESD local area network (LAN).

History	Effective Date
<p>SESDPROC-106-R3, <i>Field Measurement of Dissolved Oxygen</i>, replaces SESDPROC-106-R2</p> <p>General Corrected any typographical, grammatical, and/or editorial errors.</p> <p>Title Page Changed Chief, Enforcement and Investigations Branch from Archie Lee to Danny France. Changed Chief, Ecological Assessment Branch from Bill Cosgrove to John Deatrick. Changed Field Quality Manager from Liza Montalvo to Bobby Lewis.</p> <p>Revision History: Changes were made to reflect the current practice of only including the most recent changes in the revision history.</p> <p>Section 1.1: In the last sentence, replaced “diffusion” with “reaeration.”</p> <p>Section 1.2: Added the following statement: “Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.”</p> <p>Section 2:</p> <p>First Paragraph, Second Sentence: The phrase “taken to the field” was replaced with “utilized in the field”.</p> <p>Third Paragraph: This paragraph, which discussed a 24 hour burn in period for Clarke Cell probes, was omitted.</p> <p>Fourth Paragraph: This paragraph, which discussed Winkler Titration, was omitted.</p> <p>Section 3.1: Converted part of the first paragraph into bulleted items. Omitted all language that referred to Winkler Titration.</p> <p>Section 3.2: Omitted all language that referred to Winkler Titration.</p>	January 8, 2014
SESDPROC-106-R2, <i>Field Measurement of Dissolved Oxygen</i> , replaces SESDPROC-106-R1	February 12, 2010

SESDPROC-106-R1, <i>Field Measurement of Dissolved Oxygen</i> , replaces SESDPROC-106-R0	November 1, 2007
SESDPROC-106-R0, <i>Field Measurement of Dissolved Oxygen</i> , Original Issue	February 05, 2007

TABLE OF CONTENTS

1	General Information	5
1.1	Purpose.....	5
1.2	Scope/Application	5
1.3	Documentation/Verification.....	5
1.4	References.....	5
1.5	General Precautions.....	6
1.5.1	<i>Safety</i>	6
2	Quality Control	7
3	Field Measurement of Dissolved Oxygen.....	8
3.1	General.....	8
3.1.1	<i>Clark Cell Probes</i>	8
3.1.2	<i>Luminescent Probes</i>	8
3.2	Calibration.....	9
3.3	Maintenance	9
3.4	Conducting Field Measurement of Dissolved Oxygen.....	10
3.5	Operational Check.....	10

1 General Information

1.1 Purpose

This document describes methods and considerations to be used and observed when conducting field measurements of dissolved oxygen in surface water, treated wastewater and in gas media for specific applications (e.g., reaeration measurement).

1.2 Scope/Application

On the occasion that SESD field investigators determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another method must be used to obtain a measurement of dissolved oxygen, the alternate procedure will be documented in the field log book, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

SESD Field Branches Quality Management Plan, SESDPLAN-001, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Refer to the SESD Safety, Health and Environmental Management Program Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. When using this procedure, minimize exposure to potential health hazards through the use of protective clothing, eye wear and gloves. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

Appropriate precautions should be observed when working in and around bodies of water and on boats. Be aware of fast flowing waters, waterway obstructions such as dams, and other vessels on the water.

2 Quality Control

All dissolved oxygen meters will be maintained and operated in accordance with the manufacturer's instructions and the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). Before a meter is utilized in the field, it will be calibrated and verified, according to Section 3.2 of this procedure, to ensure it is operating properly. These calibration and verification checks will be documented and maintained in a logbook.

For in-situ measurements, an instrument warm-up period appropriate for that instrument should be provided. Consult manufacturer's documentation for appropriate warm-up time.

The ambient temperature in the immediate vicinity of the meter should be measured and recorded in the field logbook to insure the instrument is operated within the manufacturer's specified range of operating temperatures. For instruments that are deployed for in-situ measurements, the temperature of the medium being monitored should be measured and recorded in the logbook prior to deployment. *In-situ monitoring equipment may be utilized in unattended deployments where autonomous logging may preclude temperature measurement prior to deployment. Because in situ instrumentation generally has a wide range of operating temperature, the field investigator may utilize professional judgment in determining if the operating environment is suitable for unattended deployment.*

Following instrument use, an end check should be performed using one of the techniques described in Section 3.2 to quantify potential instrument drift during use.

If at any time during a field investigation, it appears that the environmental conditions could jeopardize the quality of the measurement results, the measurements will be stopped. This will be documented in the field logbook.

3 Field Measurement of Dissolved Oxygen

3.1 General

Dissolved oxygen can be defined as the volume of oxygen contained in a volume of water. The solubility of oxygen in water is dependant on the water temperature, salinity and pressure.

- As the temperature of the water decreases, the solubility of oxygen increases.
- As salinity increases, the solubility of oxygen decreases.
- As the pressure decreases (altitude increases), the solubility of oxygen decreases.

Several methods for measurement of dissolved oxygen in water are available utilizing a variety of technologies.. When measuring dissolved oxygen for compliance with the National Pollutant Discharge Elimination System (NPDES) Program, only approved methods will be used. Approved methods can be found in the Code of Federal Regulations (CFR) 40 CFR Part 136.

3.1.1 Clark Cell Probes

Clark cell probes utilize an oxygen permeable membrane that covers an electrolytic cell which consists of a cathode and an anode. The anode acts as a reference electrode. After passing through the permeable membrane, the oxygen is reduced by an applied potential voltage that is referenced to the anode. The reduction current at the cathode is directly proportional to the partial pressure of oxygen in liquid, expressed as %-air saturation. The concentration of oxygen, in mg/l, is calculated based on the %-air saturation reading and the solubility of oxygen in water at the sample temperature.

In general, sample collection using a DO probe requires only lowering the probe into the sample media and recording or logging the results. The probe should be lowered gently to prevent damage to the membrane and gently turned when initially lowered to remove any attached air bubbles. If the instrument requires the use of a stirrer, the stirrer should be turned on before recording any readings. Prior to use, the instrument should be calibrated and any manufacturer specified warm-up period should be observed.

3.1.2 Luminescent Probes

Luminescent dissolved oxygen probes employ a light emitting diode (LED) to provide incident light, which excites the oxygen-sensitive luminescent-dye molecule substrate of the sensor. After dissipation of the excitation energy, longer-wavelength light is emitted (luminescence). The magnitude of steady-state

luminescence (intensity) is measured by the sensor and is inversely proportional to the dissolved oxygen concentration.

Sample collection with this type of probe should follow the sample procedures described in the second paragraph of Section 3.1.2 for Clark Cell probes.

3.2 Calibration

Many brands of instruments are commercially available for *in-situ* measurement of dissolved oxygen using Clark cell probes and luminescent probes. The manufacturer's instruction manual should be consulted for specific procedures regarding their calibration, maintenance and use. Calibration of any measurement instrument must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate.

In general, calibrations should be conducted at temperatures and pressures as close as possible to those of the sample media for the most accurate measurements. Due to the sensitivity of dissolved oxygen measurements to changes in temperature, the temperature probe or thermistor should be verified using a NIST traceable thermometer prior to each calibration. Most dissolved oxygen meters utilize a one-point calibration which is generally performed using either water saturated air or air-saturated water. When using the water-saturated air method, the probes should be placed in a 100% relative humidity environment and the temperature and dissolved oxygen readings should be allowed to equilibrate. After equilibration, the meter should be set to read the appropriate dissolved oxygen concentration based on the temperature and barometric pressure.

When using air-saturated water for calibration, an aeration device such as an aquarium pump with a diffusion stone should be placed in a vessel containing tap water. The water in the vessel should be aerated for a minimum of one hour at a constant temperature. Saturation should be verified by placing the dissolved oxygen probe in the vessel and monitoring the temperature and dissolved oxygen readings for stabilization. Avoid placing the probe in the direct stream of air bubbles. Bubbles can accumulate on the probe surface and cause erroneous readings. Once the water is saturated, the temperature of the water and the barometric pressure can be used to determine the dissolved oxygen value. The meter can then be set to read that value.

3.3 Maintenance

Maintenance procedures vary depending on the technology utilized by each instrument and the manufacturer. The manufacturer's instruction manual should be consulted for instrument specific procedures. Following are some general guidelines for maintaining dissolved oxygen meters:

- Inspect probes for damage prior to use.

- For Clark cell probes, membranes and electrolyte solution should be changed prior to each study, when feasible.
- Battery voltages should be checked. For meters that will be deployed unattended, new or fully charged batteries should be used for each study.
- All calibration and maintenance procedures performed should be thoroughly documented.

3.4 Conducting Field Measurement of Dissolved Oxygen

Following are guidelines for conducting field measurements of dissolved oxygen:

- Except as described in specific operating procedures, dissolved oxygen measurements should if possible be conducted *in-situ*.
- When measuring DO at distinct points in the water column, the probe should be allowed to equilibrate at each location prior to recording the measurement.
- In water bodies with a great deal of flow, a weight may be attached to the probe guard or support cable to insure the probe is maintained at the proper depth.
- Insure that the measurement location is representative of conditions within the water body or reach. Avoid measurements directly below turbulent sections or still water unless these conditions represent most of the water body or reach.
- If DO measurements are conducted in saline water, the DO meter should either be capable of correcting for salinity or a separate instrument should be used to measure salinity so that the final DO measurements can be corrected.

3.5 Operational Check

A post-operation instrument verification check will be performed using one of the techniques described in Section 3.2 to quantify potential instrument drift during use. A verification check will be performed at the end of all measurements for a day or at the end of a deployment. The verification DO concentration will be measured and recorded in the field logbook prior to any instrument adjustment.

It may be appropriate to check the calibration of a DO meter periodically during the course of a day's measurements when conducting individual measurements rather than deploying an instrument. When this is done, it should be noted in the field logbook. The calibration of meters checked throughout the day maybe adjusted if drift is occurring.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: Equipment Inventory and Management

Effective Date: February 6, 2013

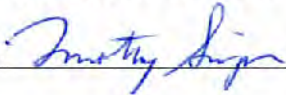
Number: SESDPROC-108-R4

Author

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Signature:



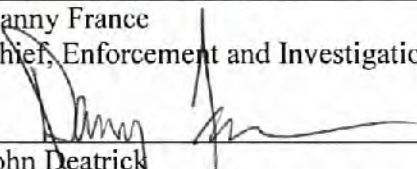
Date: 02/05/13

Approvals

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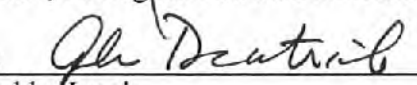


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Signature:



Date: 2/5/13

Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator (DCC).

History	Effective Date
<p>SESDPROC-108-R4, <i>Equipment Inventory and Management</i>, replaces SESDPROC-108-R3.</p> <p>General: Corrected any typographical, grammatical, and/or editorial errors.</p> <p>Title Page: Changed the EIB Branch Chief from Archie Lee to Danny France. Changed the EAB Branch Chief From Bill Cosgrove to John Deatruck. Changed the Field Quality Manager from Laura Ackerman to Bobby Lewis.</p> <p>Section 1.2: Added the following statement - Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.</p> <p>Section 1.3: Omitted references to the H: drive.</p> <p>Section 1.4.7: Defined SESD Field Equipment Tracking System.</p> <p>Section 2.4.1: Modified statement to read - Maintenance, calibration and verification requirements for equipment will be documented in the SESD Field Equipment Tracking System or equipment log.</p> <p>Section 2.4.2: Modified the statement to read - The BFEMs are responsible for placing all documentation associated with the equipment repair in the SESD Field Equipment Tracking System or appropriate maintenance section of the equipment log. Modified the statement to read - The BFEM or qualified individual is responsible for placing all documentation associated with the repair of equipment stored at the SESD laboratory in the SESD Field Equipment Tracking System or appropriate maintenance section of the equipment log.</p> <p>Section 2.5.1: All calibrations or calibration verifications performed at the SESD laboratory or Field Equipment Center will be recorded in the SESD Field Equipment Tracking System or appropriate equipment log.</p> <p>Section 2.5.2: Modified statement to read - Thermometers used during field investigations will be verified by the SESD Analytical Support Branch (ASB) or EPA contract personnel utilizing the ASB Standard Operating Procedure for the Certification of Laboratory Thermometers, SOP ASB 100G.</p> <p>Section 2.7: Modified statement to read - This will be done using the SESD Field Equipment Tracking System or by using an equipment sign-out/sign-in form.</p>	<p>February 6, 2013</p>

<p>SESDPROC-108-R3, <i>Equipment Inventory and Management</i>, replaces SESDPROC-108-R2.</p> <p>General Information Previous versions of the document will be maintained by the Document Control Coordinator (DCC). Changed from Field Quality Manager (FQM) to DCC.</p> <p>Title Page Changed EIB Branch Chief from Antonio Quinones to Archie Lee.</p> <p>Section 1.3 Changed requirement so that the DCC is responsible for ensuring the most recent version of the procedure is placed on the SESD H: drive and for maintaining records of review conducted prior to its issuance.</p> <p>Section 1.6 Added reference to Analytical Support Branch Standard Operating Procedure for the Certification of Laboratory Thermometers, SOP ASB 100G.</p> <p>Section 2.2.2 Removed requirement that the BFEM will maintain copy of all paperwork associated with the purchase of equipment.</p> <p>Section 2.3.1 Removed requirement that the BFEM send the current copy of the equipment inventory to the FQM. The BFEM will maintain the current list of equipment subject to the procedure. Modified #9 to state that the inventory list will include a reference to the manufacturer's instruction manual title.</p> <p>Section 2.3.2 Removed requirement that FQM will ensure that duplicate identification numbers are not assigned to equipment. BFEMs will be responsible for ID numbers.</p> <p>Section 2.4.2 Removed requirement that the BFEM must notify the FEC personnel via email that the repaired equipment has been returned.</p> <p>Section 2.6, #2 Acceptance record will be a copy of the signed invoice.</p>	<p>April 6, 2009</p>
<p>SESDPROC-108-R2, <i>Equipment Inventory and Management</i>, replaces SESDPROC-108-R1.</p> <p>General Changed title on cover page for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch.</p> <p>Section 1.6 Corrected form number for Load-In Form from SESDFORM-012 to SESDFORM-</p>	<p>October 19, 2007</p>

<p>011.</p> <p>Section 2.4.2 Corrected form number for Load-In Form from SESDFORM-012 to SESDFORM-011. Added last paragraph regarding adversely impacted data.</p> <p>Section 2.5 Added second sentence regarding pos-calibration verification checks for equipment. Added forth and fifth sentences regarding calibration acceptance criteria. Added second paragraph pertaining to outside calibration services.</p> <p>Section 2.3.1 Added item 9.</p>	
<p>SESDPROC-108-R1, <i>Equipment Inventory and Management</i>, replaces SESDPROC-108-R0.</p> <p>Section 1.1 Modified purpose to include equipment that may come into direct contact with the sample media and has the potential to cross contaminate samples between sampling stations</p> <p>Section 1.2 Modified scope to include microscopes, volumetric equipment and equipment that may come into direct contact with the sample media (i.e. submersible pumps) and has the potential to cross contaminate samples between sampling stations</p> <p>Section 1.3 Modified requirements for maintaining official copy of procedure.</p> <p>Section 1.5 Combined section 1.5.1 and 1.5.2. Added “maintenance” to list of manufacturer’s directions to follow. Added that a copy of the instruction manual will be provided with sampling equipment to be taken in the field.</p> <p>Section 2.1 Added equipment that comes in direct contact with sample media to list of equipment covered under the procedure. Added reference to decontamination procedures.</p> <p>Section 2.2 Renamed Section 2.2 to include equipment receipt. Added Section 2.2.2 (Receiving Equipment). Section 2.2.1 - Modified and expanded the purchase of equipment procedure. Removed reference to SESDPROC-015.</p> <p>Section 2.3 Incorporated inventory procedures from SESDPROC-104 to this section. Added Section 2.3.1 -Equipment Labeling and Marking.</p> <p>Section 2.4 Added critical spare parts will be sent out with equipment. Section 2.4.2 - Changed email notification to written notification. Included reference to SESDFORM-011. Added procedures for equipment that require software upgrades.</p>	<p>October 1, 2007</p>

<p>Section 2.5 Included visual inspection of equipment. Added reference to equipment that is not directly used for field measurement and added that calibration and measurement requirements are found in individual measurement procedures. Added maintenance procedure for microscopes and volumetric equipment. Changed title of Section 2.5.2 to include certification and verification records. Changed storage location of NIST Certificates. Defined NIST as National Institute of Standards and Technology. Added language to verify the quality of equipment. Added procedure for the verification of thermometers. Section 2.5.3 – Added that records of calibration will be maintained in equipment log books and procedure for returning equipment unsuitable for use.</p> <p>Section 2.6 Changed requirement from the original Purchase Order to a copy of the order will be maintained by the branch field equipment managers.</p> <p>Section 2.8 Removed Section from operating procedure</p> <p>Section 2.9 Removed Section 2.9 from operating procedure. Information on the glove and bottle return policy was added to Section 2.6 of SESDPROC-011-R1.</p>	
<p>SESDPROC-108-R0, Equipment and Supply Management, Original Issue.</p>	<p>May 4, 2007</p>

TABLE OF CONTENTS

1	General Information.....	7
1.1	Purpose.....	7
1.2	Scope/Application.....	7
1.3	Documentation/Verification	7
1.4	Definitions	7
1.4.1	<i>Calibration.....</i>	7
1.4.2	<i>Calibration Verification</i>	8
1.4.3	<i>Preventative Maintenance</i>	8
1.4.4	<i>Branch Field Equipment Manager</i>	8
1.4.5	<i>Qualified Individual.....</i>	8
1.4.6	<i>Equipment Log</i>	8
1.4.7	<i>SESD Field Equipment Tracking System</i>	8
1.5	Precautions.....	8
1.6	References	9
2	Methodology	10
2.1	General	10
2.2	Equipment Purchase and Receipt.....	11
2.2.1	<i>Purchasing Equipment</i>	11
2.2.2	<i>Receiving Equipment</i>	11
2.3	Equipment Inventory	12
2.3.1	<i>Inventory.....</i>	12
2.3.2	<i>Equipment Labeling and Marking</i>	12
2.4	Equipment Maintenance.....	13
2.4.1	<i>Documentation</i>	13
2.4.2	<i>Repair and Re-certification for Use</i>	13
2.4.3	<i>Required Maintenance.....</i>	14
2.4.4	<i>Record Keeping</i>	14
2.5	Equipment Calibration	14
2.5.1	<i>Documentation</i>	15
2.5.2	<i>Traceability, Certification, and Verification Records.....</i>	15
2.5.3	<i>Equipment Performance Checks</i>	16
2.6	Records.....	17
2.7	Equipment Sign-out/Sign-in	17

Contents

1 General Information

1.1 Purpose

This document describes the procedures necessary to demonstrate the operational status and inventory of equipment used for field measurement activities and equipment that comes into direct contact with the sample media and has the potential to cross contaminate samples between sampling stations.

1.2 Scope/Application

This procedure covers the approaches and documentation used for the purchase, maintenance, calibration, verification and inventory of equipment used for direct field measurement activities. This procedure includes the maintenance and use of microscopes, volumetric equipment and equipment that may come into direct contact with the sample media (i.e. submersible pumps) and has the potential to cross contaminate samples between sampling stations. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 Definitions

1.4.1 Calibration

The set of operations which establish, under specified conditions, the relationship between values indicated by a measuring instrument or measuring system and corresponding known values. The results of a calibration permit the estimation of errors associated with the measurement equipment.

1.4.2 Calibration Verification

Provides a means of determining that deviations between measured values and known values are within the limits of error defined during calibration. The results provide an indication that the instrument/system is working properly.

1.4.3 Preventative Maintenance

A program of routine actions such as cleaning, lubrication, adjusting, or testing to keep equipment ready for use. The most important effect of a preventative maintenance program is to ensure measurement system reliability.

1.4.4 Branch Field Equipment Manager

Staff, designated by management, who are responsible for ensuring that the procedures for Equipment Inventory and Management are followed. At least one Branch Field Equipment Manager (BFEM) will be designated for the Enforcement and Investigations Branch (EIB) and the Ecological Assessment Branch (EAB).

1.4.5 Qualified Individual

Individual who has received on the job training and has experience working with specific measurement instruments.

1.4.6 Equipment Log

Notebook, log book or electronic file that contains a copy of the purchase order, if available, as well as, maintenance, calibration, verification records, performance checks, correction factors and sign-out/sign-in records. Equipment logs will be established for all equipment used for field measurement activities or equipment that comes into direct contact with the sample media and has the potential to cross contaminate samples between sampling stations. The development of equipment logs is the responsibility of the Branch Field Equipment Managers.

1.4.7 SESD Field Equipment Tracking System

Electronic database used to sign-out/sign-in sampling equipment, track equipment use history, and maintain equipment availability status.

1.5 Precautions

To ensure the safe and reliable operation of equipment, the manufacturers' directions for transport, cleaning, decontamination, storage, maintenance and operation of equipment

will be followed. When possible, a copy of the instruction manual will be provided with sampling equipment to be taken in the field. In addition, field activities will be conducted in accordance to the SESD Safety, Health and Environmental Management Program (SHEMP).

Handling of contaminated equipment is addressed under the following operating procedures: Field Equipment Cleaning and Decontamination at the Field Equipment Center (SESDPROC-206) and Field Equipment Cleaning and Decontamination (SESDPROC-205).

1.6 References

American National Standard ANSI/ASQC M1-1996, *Calibration Systems*

American National Standard ANSI/NCLS Z2540-1-1994, *Calibration Laboratories, and Measuring and Test Equipment - General Requirements*

SESD Safety, Health and Environmental Management Program (SHEMP) Manual, most recent version.

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, most recent version.

SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, SESDPROC-206, most recent version.

SESD Operating Procedure for Competency and Proficiency Testing, SESDPROC-006, most recent version.

SESD Operating Procedure for Purchasing of Services and Supplies, SESDPROC-015, most recent version.

SESD Operating Procedure for Control of Nonconforming Work, SESDPROC-019, most recent version.

FEC Loan-In Form, SESDFORM-011, most recent version.

Analytical Support Branch Standard Operating Procedure for the Certification of Laboratory Thermometers, SOP ASB 100G.

2 Methodology

2.1 General

Equipment (hardware and related software) used for field measurement activities and equipment that comes into direct contact with the sample media and has the potential to cross contaminate samples between sampling stations will meet quality requirements identified for each piece of equipment. Important factors in establishing quality requirements include the parameters to be measured and the sensitivity and specificity of the detection system used. Quality requirements must include ensuring that equipment is ready for use. Specifically:

1. SESD will have adequate equipment to conduct measurement activities.
2. Equipment used for field measurement activities will be capable of achieving the accuracy and precision required by the measurement objectives.
3. When SESD is required to use environmental data collection equipment outside its permanent control, it will ensure that all relevant SESD criteria in this procedure are met.
4. Field measurement equipment used by SESD will be secured at all times, as feasible, based on field conditions.
5. Operating instructions and/or manuals from the manufacturer will be available for each piece of equipment, when possible.
6. Field investigators will only operate equipment for procedures which they are authorized per SESDPROC-006 to perform.
7. Equipment used for field measurements will be handled, transported, shipped, stored and operated in a manner that prevents damage, gross contamination and deterioration. Equipment will be handled and maintained in accordance to the manufacturer's operating instructions. Decontamination of equipment will be in accordance to procedures described in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) and Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206).

2.2 Equipment Purchase and Receipt

2.2.1 Purchasing Equipment

Equipment covered in this procedure is purchased using procurement requests (PR) or purchase card orders and can be initiated by the BFEM or designee. The initiator of a purchase is responsible for preparing a PR or purchase card order that specifies the correct name of the item, the relevant quality criteria of the item, the item number, a quote or price, possible sources and any other associated information that helps identify the correct item to be purchased. The sources identified must be capable of providing the equipment according to the defined specifications. A copy of the purchase order or purchase card order will be maintained in the equipment log. Once the order has been prepared, the information is forwarded to the appropriate BFEM for origination of the purchase and management will authorize and sign the order and forward it to the purchasing official. The purchasing official will ensure the order is processed according to the information on the PR or purchase card order.

2.2.2 Receiving Equipment

Anyone can accept delivery of equipment orders. For equipment purchases received at the SESD laboratory, the BFEM or initiator of the purchase will inspect the items to ensure they comply with the PR or purchase card order. If the items are acceptable, the BFEM or initiator will sign and date the invoice that accompanied the order and any other required documentation and forward it to the purchasing official. The purchasing official will maintain a file of all SESD purchases.

For purchases received at the Field Equipment Center, contract personnel will inspect the items to ensure they comply with the PR or purchase card order. If the items are acceptable, the contract personnel will sign and date the invoice that accompanied the order and any other required documentation and forward it to the appropriate BFEM. The original paperwork (signed invoice) will be forwarded to the purchasing official.

If any equipment is found to be unsuitable for use, the BFEMs will document the problems and any action taken to correct that problem. The documentation will include a description of the item, the deficiency and the vendor. The BFEM will also compile all occurrences of unsuitable consumables, supplies or services and determine what further corrective action may be necessary and notify the Field Quality Manager if deemed necessary.

2.3 Equipment Inventory

2.3.1 Inventory

The BFEMs are responsible for maintaining a current equipment inventory list for their specific areas. The BFEMs will maintain a central, comprehensive list of all field measurement and sampling equipment subject to this procedure. Field measurement equipment, including sampling equipment that comes into contact with the sample media and has the potential to cross contaminate samples between stations (i.e., submersible pumps and vacuum chambers), will be included in this inventory. The equipment inventory list for each instrument or piece of equipment will include:

1. A description of the property and software, if applicable (e.g., pH meter, dissolved oxygen meter, etc.).
2. Manufacturer or vendor name
3. Model number
4. Serial number or other manufacturer identification number
5. A unique identifier, known as the SESD ID Number. These identifiers will be assigned by the BFEM.
6. Storage location (e.g., Field Equipment Center, SESD Laboratory)
7. Date received and/or date placed in service, where available.
8. Status of the equipment (e.g., surplus, damaged, etc.)
9. A reference to the manufacturer's instruction manual title.

2.3.2 Equipment Labeling and Marking

Prior to being placed into the equipment inventory, all equipment covered under this procedure will be inventoried and labeled with a unique SESD identification number. The identification number will be assigned only to the identified equipment and will not be reused if the meter is excised or disposed. Equipment that is no longer used or cannot be repaired will be removed from the inventory.

Equipment received after the effective date of this procedure will be assigned a number that includes the date the equipment was received followed by a sequential number starting at 01 (e.g., 031407-01, 031407-02, etc.). BFEMS will ensure that duplicate SESD ID Numbers are not assigned to equipment.

Equipment received before the effective date of this operating procedure will be assigned a number that will incorporate all or part of the serial number or other manufacturer number as the unique SESD ID number.

2.4 Equipment Maintenance

Maintenance will consist of preventative care and corrective repair. Both approaches should be used to keep equipment in working order. Each item of field measurement equipment will be checked by a qualified individual prior to use. Critical spare parts, which cannot be easily obtained while in the field, will be sent out with the equipment during field investigations.

Equipment maintenance will include software upgrades for certain instruments. SESD management will assign staff to serve as a point of contact for equipment, such as GPS or Sondes that may require periodic software upgrades in order to collect field measurements. The point of contact will ensure that software upgrades are conducted on all equipment. The point of contacts will maintain a list of the equipment which will include the serial number, SESD ID Number, software version, software upgrade dates and the storage location of the equipment. The point of contact will provide the information to the appropriate BFEM to be included in the equipment inventory.

2.4.1 Documentation

The BFEMs are responsible for the development of equipment logs. Maintenance, calibration and verification requirements for equipment will be documented in the SESD Field Equipment Tracking System or equipment log.

2.4.2 Repair and Re-certification for Use

Equipment known or suspected to be defective will be taken out of service and clearly labeled, preferably with a red tag, until it has been repaired and shown by calibration, verification or testing to function properly. When equipment is tagged in the field as defective, the SESD project leader will notify the appropriate BFEM in writing, either by email or by using a load-in form (SESDFORM-011), that equipment requires repair.

For equipment maintained at the FEC which is routinely handled and maintained by contract personnel, the BFEM will be responsible for having the equipment repaired. Once repaired, the BFEM will notify the FEC contract personnel that the equipment has been returned. The contract personnel will ensure that the equipment is functioning properly prior to it being used for field investigations. The BFEMs are responsible for placing all documentation associated with the equipment repair in the SESD Field Equipment Tracking System or appropriate maintenance section of the equipment log. This documentation includes written notifications regarding the equipment repair and any information provided from the manufacturer which describes the defect and the repair.

For equipment stored at the SESD laboratory, or stored at the FEC and not routinely handled and maintained by contract personnel, the BFEM or a designated qualified individual will be responsible for having the equipment repaired. Once repaired, the BFEM or qualified individual will ensure the equipment is functioning properly prior to it being used for field investigations. The BFEM or qualified individual is responsible for placing all documentation associated with the repair of equipment stored at the SESD laboratory in the SESD Field Equipment Tracking System or appropriate maintenance section of the equipment log. This documentation includes the written notifications regarding the equipment repair and any information provided which describes the defect and the repair.

If there is the potential that data collected with defective equipment were adversely impacted, the BFEM will notify the appropriate Branch Quality Assurance Officer (QAO). The QAO will notify affected management and The Field Quality Manager and the issue will be handled according to the SESD Operating Procedure for Control of Nonconforming Work (SESDPROC-019).

2.4.3 Required Maintenance

The BFEMs are responsible for ensuring that equipment maintenance is performed in-house or by an outside service.

2.4.4 Record Keeping

Records of all maintenance, service, repairs and histories of any damage, malfunction or modification of field measurement equipment will be maintained in the equipment logs. The record will describe hardware and software changes and/or updates and show the dates when these occurred.

2.5 Equipment Calibration

Prior to being used in the field, measurement equipment will be visually inspected and a calibration, calibration verification or performance check will be conducted to ensure it is in working condition. A post-operation instrument verification check will be performed using the appropriate standard(s) at the end of the day or after all measurements have been taken for a particular period of operation. When practicable, equipment requiring calibration will be labeled to indicate the status of calibration and the date when re-calibration is due. Calibration acceptance criteria based on data quality objectives will be established on a case by case basis for each field study. Project Leaders will be responsible for ensuring the acceptance criteria are met. Calibration and measurement requirements are found in individual measurement procedures.

When equipment or reference standards are calibrated by an outside calibration service, SESD will ensure the vendor is ISO 17025 accredited or has some other equivalent means for demonstrating competence, measurement capability and traceability.

All reasonable measures will be taken to safeguard equipment from adjustments which would invalidate the measurements following calibration of the equipment. Field investigators will handle, transport and store equipment in accordance to manufacturer recommendations. Periodic calibration or performance checks will be conducted during field investigations to ensure that equipment calibration has not been adversely impacted between measurement locations.

For equipment that is not directly used for field measurement, visual examination, safety checks or, if appropriate, performance checks, will be conducted by the FEC contract personnel or the project leader to ensure the working condition of the equipment. Microscopes, including attachments, will be cleaned and serviced as needed prior to use.

Volumetric equipment will be maintained by visual examination and cleaning at the Field Equipment Center. Performance checks will be conducted by the FEC contract personnel or the project leader prior to initial use and at intervals depending on the frequency of use.

2.5.1 Documentation

Records for equipment that is sent off-site for calibration recertification will be maintained in the equipment log books. All calibrations or calibration verifications performed at the SESD laboratory or Field Equipment Center will be recorded in the SESD Field Equipment Tracking System or appropriate equipment log.

All calibrations or calibration verifications performed in the field will be recorded in the project specific logbook(s) by the individual conducting the calibration or verification and stored in the associated project file. Calibration of equipment conducted by SESD field investigators and other qualified personnel will be conducted in accordance with the manufacturer's recommendations

When calibrations require correction factors, the correction factors will be documented in instrument operating procedures, equipment logs and written methods (field logbooks).

2.5.2 Traceability, Certification, and Verification Records

The program for the calibration or calibration verification of equipment must ensure that, where the concept is applicable, all significant measurements are traceable through certificates of calibration held by SESD, and/or to National

Standards of Measurement. National Institute of Standards and Technology (NIST) traceable standards will be used, if available. For equipment maintained at the FEC and sent off-site for calibration, certificates or other records of calibration will be maintained in a central file at the FEC. Additionally, NIST traceable documentation for buffers, standards, calibration gases or reference materials will be maintained in a central file at the FEC.

For equipment maintained at the SESD laboratory and sent off-site for calibration certificates or other records of calibration will be maintained in the lab where the equipment is stored. Additionally, NIST traceable documentation for buffers, standards, calibration gases or reference materials will be maintained in a central file at the SESD laboratory with the equipment.

SESD will maintain records of actions taken to verify the quality of equipment whose properties could affect the quality of sampling, measurement and related activities. Examples would be thermometer, calibration standard or buffer verification. Thermometers used during field investigations will be verified by the SESD Analytical Support Branch (ASB) or EPA contract personnel utilizing the ASB Standard Operating Procedure for the Certification of Laboratory Thermometers, SOP ASB 100G. The SESD ASB is accredited by the National Environmental Laboratory Accreditation Conference.

Critical reference materials such as reagents and consumable materials that affect the quality of tests and/or calibrations will be verified according to the procedures described in the SESD Operating Procedure for Purchasing Services and Supplies, SESDPROC-015.

2.5.3 Equipment Performance Checks

New equipment will be calibrated or verified by qualified personnel to verify that it is adequate to perform its intended function, and calibrated before release for use in the field. Records of calibration or verification will be maintained in the equipment log books. If any equipment proves to be unsuitable for use, the BFEMs will document the issue, notify the purchasing official and return the deficient equipment to the vendor.

If equipment leaves the direct control of SESD for a period of time, the BFEMs will ensure that the function and, where necessary, the calibration status of the equipment is checked and shown to be satisfactory before the equipment is returned to service.

2.6 Records

The BFEMs are responsible for maintaining a current equipment inventory list for their specific programs. The FQM will maintain a central, comprehensive list of all field measurement and sampling equipment subject to this procedure.

The BFEMs are responsible for creating equipment logs for all equipment in their specific program used for field measurement activities. Additionally, they are responsible for auditing and maintaining equipment maintenance, calibration and verification records in the equipment logs. These records must be made available to SESD personnel prior to their use for field measurement.

These records will include:

1. A copy of the Purchase Order, if available, or other record showing item received and date placed into service
2. Acceptance inspection record of the equipment (copy of signed invoice)
3. Maintenance, calibration and verification logs (including software if applicable)
4. Identification of individuals accessing maintenance, calibration and verification logs
5. Calibration certificates for manufacturers calibrations (if applicable)
6. Fundamental calibration or any other performance checks information
7. Calibration verification record (showing standards/reference materials used)
8. Calibration correction factors, if applicable
9. Records of handling, transportation and storage of equipment
10. Complaint log (if applicable)
11. A record of the most recent version of firmware or software for the equipment.

2.7 Equipment Sign-out/Sign-in

All measurement equipment will be signed-out prior to use in the field and signed-in following use in the field. This will be done using the SESD Field Equipment Tracking System or by using an equipment sign-out/sign-in form. Each form is specific to the type of measuring equipment being checked out and includes calibration or verification data, as well as, project name and project leader information.

FEC contract personnel will primarily be responsible for equipment sign-out and sign-in at the FEC. However, in their absence, it is the project leader's responsibility to sign out the measurement equipment. For measurement equipment that is not routinely handled or maintained by contract personnel or equipment stored at the SESD laboratory, the project leader or field investigator using the equipment is responsible for checking the equipment out and in.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: Global Positioning System

Effective Date: April 20, 2011

Number: SESDPROC-110-R3

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Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
<p>SESDPROC-110-R3, <i>Global Positioning System</i>, replaces SESDPROC-110-R2</p> <p>Cover Page: The EIB Branch Chief was changed from Antonio Quinones to Archie Lee. The FQM was changed from Laura Ackerman to Liza Montalvo.</p> <p>Revision History: Changed Field Quality Manager to Document Control Coordinator.</p> <p>Section 1.2: Added the following statement: Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.</p> <p>Section 1.3: Omitted the reference to the H: drive of the LAN. Changed Field Quality Manager to Document Control Coordinator.</p>	April 20, 2011
<p>SESDPROC-110-R2, <i>Global Positioning System</i>, replaces SESDPROC-110-R01</p> <p>Title Page Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove's title from Acting Chief to Chief.</p> <p>Section 1.4 Alphabetized and added a reference.</p>	November 1, 2007
<p>SESDPROC-110-R1, <i>Global Positioning System</i>, replaces SESDPROC-110-R0</p> <p>This revision reflects the following changes:</p> <p>General Extensive rewrite with additional information added to most sections. Brian Striggow added as coauthor.</p> <p>Section 2.1 Rewritten description of GPS system. Descriptions of "GPS Accuracy Factors" and "Differential GPS" isolated as separate subsections.</p>	October 1, 2007

<p>Section 2.2 This Section, titled “Requirements for Locational Information” inserted. Supplies guidance on accuracy requirements for various study types and potential means used to obtain requisite accuracy. Subsection on datums and data formats included.</p> <p>Section 2.3 “Quality Control Procedures”, previously numbered as Section 2.2. Most information in this section moved to Section 2.1 discussion of Accuracy Factors and Section 2.4 discussion of specific receivers. Explicit statement added that no specific GPS quality control procedures are required for most SESD studies.</p> <p>Section 2.4 Information added to sections on Trimble and Garmin receivers.</p> <p>Section 2.5 Issue of electronic data-logging addressed. Storage of electronic data records addressed.</p>	
<p>SESDPROC-110-R0, Global Positioning System, Original Issue</p>	<p>March 22, 2007</p>

TABLE OF CONTENTS

1	General Information	5
1.1	Purpose.....	5
1.2	Scope/Application	5
1.3	Documentation/Verification.....	5
1.4	References	6
2	Methodology	7
2.1	General.....	7
2.1.1	<i>GPS Description</i>	<i>7</i>
2.1.2	<i>GPS Accuracy Factors</i>	<i>8</i>
2.1.3	<i>Differential GPS.....</i>	<i>9</i>
2.2	Requirements for Locational Information	10
2.2.1	<i>Data Uses</i>	<i>10</i>
2.2.2	<i>Datums and Data formats</i>	<i>12</i>
2.3	Quality Control Procedures	13
2.4	Special Considerations.....	13
2.4.1	<i>Special considerations for the use of Trimble® Mapping Grade Receivers</i>	<i>13</i>
2.4.2	<i>Special considerations for the use of Garmin® General Use Grade Receivers</i>	<i>15</i>
2.5	Records.....	16

Contents

1 General Information

1.1 Purpose

This document describes the Global Positioning System (GPS) and procedures, methods and considerations to be used and observed when using GPS to record location data in the field. Guidance is provided on accuracy requirements for various uses of location data and potential means to obtain the requisite accuracy. This document contains direction developed solely to provide internal guidance to SESD employees.

1.2 Scope/Application

The procedures contained in this document are to be used by SESD field investigators when using the Global Positioning System to obtain the geographical coordinates of sampling locations and/or measurements during field investigations. In SESD investigations, GPS is the preferred means of collecting horizontal location information. In most cases the accuracy of GPS is unsuitable for collection of elevation data.

On the occasion that SESD field personnel determine that any of the procedures described in this section cannot be used to obtain the required coordinate information and alternate procedures are employed, the alternate procedure will be documented in the field log book, along with a description of the circumstances requiring its use. GPS users must be currently qualified as proficient in the operation of the specific GPS equipment to be used. The manufacturer's operation manuals should be used for detailed information on the use of specific GPS equipment. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

Rand Corporation, The Global Positioning System, Assessing National Policies, Appendix B, GPS History, Chronology, and Budgets, 1995.

SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version.

Trimble® Navigation Limited, Mapping Systems General Reference, Revision B, 1996.

USEPA, Global Position Systems – Technical Implementation Guidance, Office of Environmental Information (EPA/250/R-03/001), 2003.

USEPA, GIS Technical Memorandum 3. Global Positioning Systems – Technology and It's Application in Environmental Programs, Research and Development (PM-225). EPA/600/R-92/036, 1992.

USEPA, Locational Data Policy, Office of Information Resources Management, IRM Policy Manual 2100 Chapter 13, 1991.

2 Methodology

2.1 General

2.1.1 GPS Description

The Navigation Satellite Time and Ranging (NAVSTAR) Global Positioning System (GPS) is a worldwide radio-navigation system created by the U. S. Department of Defense (DOD) to provide navigation, location, and timing information for military operations. System testing using a limited number of satellites began in 1978 with the system being declared fully operational in 1995. The system was declared available for civilian uses in the 1980s and has seen burgeoning civilian application for navigation and mapping.

The GPS system consists of three basic elements: the space segment, control segment, and user segment. The space segment consists of the constellation of up to 24 active NAVSTAR satellites in six orbital tracks. The satellites are not in geo-synchronous orbit and are in constant motion relative to a ground user. The control segment consists of several ground stations that serve as uplinks to the satellites and that make adjustments to satellite orbits and clocks when necessary. The user segment consists of the GPS receiver which will typically consist of an antenna, multi-channel receiver, and processing unit.

For the purposes of this document, the user segment GPS receivers may be loosely grouped into Recreational and Navigational receivers (henceforth referred to as General Use receivers), Mapping Grade receivers, and Survey Grade receivers.

- Most General Use grade receivers are available on the retail market to consumers for a variety of applications including boating, hiking, and automotive navigation. They display an instantaneous reading of position and are generally not optimized for data collection. Waypoints containing instantaneous position fixes can often be stored and downloaded. The accuracy of these receivers is adequate for many environmental applications.
- Mapping Grade receivers are used for applications such as resource management and Geographical Information System (GIS) feature collection. The receivers are capable of averaging multiple position fixes for greater accuracy and then data-logging the results with sufficient information to post-correct the positions as described below. The potential accuracy that can be achieved may be better than one meter.

- Survey Grade receivers can provide accuracy at the centimeter level by using long occupation times and special techniques for receiver use and data processing. Survey Grade receivers are not used by SESD in field investigations.

GPS receivers derive positions by simultaneously measuring the distance (range) to several satellites in precisely known orbits, and using triangulation of the ranges to calculate a unique position for the receiver. The range to each satellite is determined by precisely measuring the transit time of radio signals broadcast from the satellites.

2.1.2 GPS Accuracy Factors

The accuracy of the basic GPS system is approximately 15m. GPS accuracy can be affected by a number of factors including the Selective Availability feature, atmospheric delays, satellite clock and orbit errors, multipath signals, signal strength, and satellite geometry relative to the user.

In the early GPS implementation, the DOD used a feature known as Selective Availability (SA) to degrade the quality and subsequent accuracy of the GPS signals to non-DOD users. With Selective Availability enabled, accuracy of position fixes could be as poor as 100m without the use of differential correction techniques described below. Currently there is no SA limitation in accuracy in place with a stated Executive Branch intention to not return to the use of the SA signal degradation.

As satellites move in their orbits and some signals are blocked by obstructions, the geometry of the available satellite signals relative to the user will constantly change. When the satellites with available signals are clustered closely together in the sky, small errors in range will result in large errors in reported position. Conversely, when the satellites are distributed more broadly across the sky, the resultant position errors will be at their minimum. The general measure of this phenomenon is Dilution of Precision (DOP), which may be represented as Position Dilution of Precision (PDOP), or more specifically for geographical coordinate collection, Horizontal Dilution of Precision (HDOP). Mapping and Survey Grade receivers generally can calculate and display DOP and allow the user to limit logging to times when the higher potential accuracy conditions of low DOP prevail. General Use receivers may display DOP and use DOP with other factors to estimate a general accuracy figure. DOP may range from approximately 2 to 50, with high quality work usually requiring a HDOP of less than 4-6.

Signal strength and multipath signals relate to the strength and quality of the signal reaching the receiver antenna. Signal attenuation by the atmosphere, buildings, and tree cover limit the accuracy of the ranges obtained. The measure of signal strength is Signal to Noise Ratio (SNR), generally measured in decibels(db). Most receivers of any grade will display the SNR of the satellite signals in a bar graph or table. Mapping Grade Receivers generally allow the user to specify a minimum signal strength for the use of a satellite signal (commonly 2-15db). Poor signal strength can be resolved by waiting for satellite locations to change or moving the receiver location. Multipath signals result

from portions of the satellite signal bouncing off terrain, structures, or atmospheric disturbances, resulting in a degraded total signal. Higher quality Mapping Grade receivers may be capable of rejecting the stray multipath signals, such as Trimble® receivers using Everest™ technology.

2.1.3 Differential GPS

Selective Availability, clock errors, and orbital errors affect all GPS users and atmospheric delays affect all users over a relatively wide region. A second GPS receiver in the same general area as the user will experience the same errors from these sources as the user's receiver. Consequently, correction factors from a remote station at a known location can be applied to the user's receiver in a process known as Differential GPS (DGPS). DGPS can be applied in real-time using additional radio signals, or after the collection event by a method called post-correction.

Real-time DGPS uses established networks of base stations at precisely surveyed locations. The US Coast Guard operates a system of 80 base stations which became fully operational in 1999. The range corrections are broadcast on marine radiobeacon frequencies, with redundant coverage of most of the US coastline and the Mississippi River. There is near complete single beacon coverage of most of the internal US, but there are known gaps in coverage in both EPA Region 4 and the US as a whole. The system is sometimes referred to using the more general term DGPS or in nomenclature referring to the beacon-based nature of the system. Beacon-based DGPS is implemented primarily in Navigational and Mapping Grade receivers.

Real-time DGPS can also be implemented with a Space Based Augmentation System (SBAS). The most common SBAS currently used in the United States is the Wide Area Augmentation System (WAAS), developed by the Federal Aviation Administration to meet the additional demands on GPS for aircraft navigation. The WAAS network of base stations collects information on satellite clock errors, orbital errors, and atmospheric conditions. The error information is transferred to satellites in geo-synchronous orbits and subsequently broadcast to suitably equipped GPS receivers on frequencies compatible with the GPS range signals. While beacon-based DGPS passes range corrections to the receivers, WAAS communicates a model for the errors which is capable of providing more accurate corrections. Current Mapping Grade receivers will likely use WAAS with or without the option of beacon-based DGPS. All but the least expensive General Use receivers are generally equipped with WAAS differential correction capability.

Post-Corrected DGPS is generally accomplished by downloading the receiver survey files to a desktop or laptop computer and then retrieving correction files for the same time period (generally via the internet) from an established base station in the area of the survey. Post-processed accuracy improves with proximity of the base station to the surveyed locations and base station data should be used from a station within 300km of the site surveyed. The survey positions are processed by application software and a new

set of positions is generated using the correction data. The capability for post-processed differential correction is generally limited to Mapping Grade and Survey Grade receivers.

Various factors limit GPS accuracy in the vertical plane to approximately half of that obtainable in the horizontal plane, i.e., if a location fix is accurate to 3 m in the horizontal plane, it may only be accurate to 6 m in the vertical plane. Since relatively high accuracy is usually required for the uses of elevation data, GPS is rarely used to obtain and report elevations.

2.2 Requirements for Locational Information

2.2.1 Data Uses

Locational information can serve many purposes in an environmental investigation, a few of which are listed below:

1. Providing an unambiguous means to identify facilities or sampling plats.
2. Providing locational information to key analytical data in a GIS based data archiving system to the original sampling locations.
3. Differentiating watersheds.
4. Providing information to calculate extents and volumes of contamination.
5. Providing a means to relocate the media represented by samples for removal or treatment.
6. Providing information to prepare presentation graphics of sampling locations.

Depending on the specific uses for the data and the type of work being performed, there will be different needs for the accuracy of the locational data. Studies where a sample represents a large area of relatively homogeneous material would not require the same accuracy as the location of a permanent monitoring well. Below are broad guidelines for the accuracy that might be required for different applications.

Desired Accuracy	Application
100 m	Open ocean work where sample is presumed to be representative of a large area
20 m	Open water work (lakes or estuaries) where sample is presumed to be representative of a large area
10 m	Stream and river work where samples are presumed to be broadly representative of a reach
5-3 m	Stream work where samples are representative of a specific narrowly defined section

10 m	Air Monitoring Stations
10 - 3 m	Microscale air monitoring
3 - 1 m	Permanent monitoring wells
1 m	Locations of 'Hot Spots' destined for removal of limited areal extent
3 - 1 m	Locations of Temporary groundwater wells in plumes requiring narrow delineation
3 m	Locations of Temporary groundwater wells in broad plumes
3 m	Locations of environmental samples with sample spacing >20 m
5 m	Locations of environmental samples with sample spacing >60 m
200 - 20 m	Coordinates describing a facility where mobile waste units are sampled
30 - 3 m	Locations of industrial process areas or NPDES permitted facilities where the sampling locations are described in field notes relative to the process or site features

Specific demands of a study may drive increased or decreased requirements for accuracy. The preferred means of locational data collection for most studies will be GPS, although alternate means are permissible if they meet accuracy requirements. The following table indicates the accuracy that may be expected from various means of establishing coordinates.

Accuracy	Description
200 - 50 m	Map Derived, coarse work
40 - 20 m	Map Derived, fine work or using GIS with digital imagery
15 m	General Use Grade GPS, w/o WAAS
5 m	General Use Grade GPS, w/ WAAS or beacon corrections
10 m	Mapping Grade GPS, no corrections, averaged readings,
3 m	Mapping Grade GPS w/ differential correction, averaged readings
1 m	Mapping Grade GPS w/ differential correction, controlled DOP and SNR, averaged readings
10 cm	Surveying Grade GPS or optical surveying (dependent on baseline length)

Accuracy is a term used to describe the degree of conformity of a measurement. In GPS, accuracy is usually specified as an estimate of the radius from the measured coordinates that is likely to include the actual coordinates. The estimate is based on several standard deviations probability of including the actual point in the measurement. As such, it is recognized that some measurements will fall outside of the specified accuracy. For the purposes of SESD GPS work, the nominal accuracy figures derived from manufacturer's literature for specific operating conditions, displayed by the receiver at the time of feature collection, or output from processing software will be taken at face value.

2.2.2 Datums and Data formats

In general, a datum is a reference from which other measurements are taken. In the development of surveying systems by civil entities, different datums were used as base references that will result in differing coordinates for the same location. A GPS receiver will generally display coordinates in a number of different user-selected datums. **Unless there are specific requirements on a project, all SESD work should be conducted using the WGS84 datum.** Alternatively, the nearly equivalent NAD83 datum may be used if WGS84 is unavailable as a receiver option. If an alternate coordinate system is used where coordinates are obtained and recorded in field logbooks, the use of the alternate coordinate system should also be noted in the logbook.

The SESD Data Archival and Retrieval System (DART) requires that coordinates for sample locations be entered in the WGS84 datum and dd.dddddd format. Unless specific project requirements dictate otherwise, all coordinates explicitly stated in reports will be in WGS84 format and in all cases the datum used will be specified.

There is no SESD policy on significant digits for GPS information, and accuracy should not be implied from the presence of significant digits in reported coordinates. However, good scientific practice should be followed in the presentation of locational information in order that useful information not be truncated or a higher degree of accuracy implied. The following table shows the incremental distance in latitude represented by the least significant digit for various coordinate formats:

dd.dddddd°	Approximately 4" or 10 cm
dd.ddddd°	Approximately 44" or 1.1 m
dd.dddd°	Approximately 36' or 11 m
dd°mm'ss"	Approximately 100' or 30 m
dd°mm'ss.x"	Approximately 10' or 3 m
dd°mm'ss.xx"	Approximately 1' or 30 cm
dd°mm.xxxx'	Approximately 7" or 18 cm
dd°mm.xxx'	Approximately 6' or 1.8 m
dd°mm.xx'	Approximately 60' or 18 m

2.3 Quality Control Procedures

By nature of its origin in the DOD and recent application to aircraft navigation, the GPS is designed for high reliability. GPS failures resulting in an incorrect reading beyond the bounds of known errors are so rare that the possibility can be ignored for most SESD studies. If a study requires the verification of receiver function, this can be accomplished by verifying that a receiver displays the correct position while occupying a known benchmark.

2.4 Special Considerations

The application will dictate the type of receiver used. There are several specific considerations for the use of the various receiver types.

2.4.1 Special considerations for the use of Trimble® Mapping Grade Receivers

The suggested settings for Trimble® receivers are:

1. Position mode: Overdetermined 3D (or manual 3D if only 4 satellites are visible)
2. Elevation mask: 15 degrees
3. PDOP: 6
4. Signal to noise ratio (SNR) mask: 6
5. Dynamics code: Land
6. Logging interval: 1 second for points, 5 seconds for lines and areas
7. Audible click: Yes
8. Log DOP data: Yes
9. Coordinate System: Geographic Datum WGS84

When using Trimble® Geoexplorer receivers, the option of using the accuracy „slider bar“ display may be used to consolidate many of the above parameters. The slider bar position must be positioned at midscale or towards the „Precision“ end of the scale to achieve 1m accuracy work. When conducting work requiring less accuracy, the slider should be initially positioned midscale and may be adjusted toward the Production“ end of the scale if the higher level of accuracy can not be maintained.

The receivers may be configured to force real-time position correction. The use of real-time correction reduces the risk of being unable to obtain appropriate post-correction files, but may limit productivity when differential signals cannot be

received or are intermittent. The use of forced real-time vs. post-correction is an operator decision. If the positions are not to be downloaded and the accuracy of differential correction is required, it will be necessary to force real-time correction.

Trimble® receivers at SESD contain a data dictionary that can facilitate the management of GIS data. If the COC_GIS dictionary is selected at the time of file creation, SESD standard media codes can be assigned to features at the time of logging that will accompany the data through the download process. The use of the COC_GIS data dictionary can simplify the management of the data when processed in a GIS system or when entered into the DART system.

The logging interval of most Trimble® GPS receivers defaults to a 5 second interval. This may be changed to a 1 second interval to expedite feature collection. A point feature should have a minimum of 36 positions logged to obtain the additional accuracy afforded by averaging positions. After a minimum of 36 positions are logged and the feature is closed, the averaged position can be obtained by selecting the feature on the „Map“ screen. The averaged position should always be the one entered into field notebooks.

Some Trimble® receivers may only display data in dd°mm“ss.sss” format. If the coordinates are downloaded and processed through Pathfinder Office software, they can be output in the dd.dddddd (or any other) format. If the coordinates were only recorded in field logbooks, they can be converted to decimal degrees as follows:

Converting to decimal degrees (dd.dddddd) from degrees°minutes“seconds” (dd°mm“ss.sss”):

$$\text{dd.dddddd} = \text{dd} + (\text{mm}/60) + (\text{ss.sss}/3600)$$

Example: Convert 33°28“45.241” to decimal degrees

$$33 + (28/60) + (45.241/3600) = 33.479236$$

The reverse conversion is accomplished as follows:

Converting to degrees°minutes“seconds” from decimal degrees

Starting with dd.dddddd

Multiply .dddddd by 60 to obtain mm.mmmm

Multiply .mmmm by 60 to obtain ss.sss

Then dd°mm“ss.sss” = dd & mm & ss.sss

Example: Convert 33.479236 to dd°mm“ss.sss” format

Multiply .479236 by 60 to obtain 28.7540 (mm.mmmm)

Multiply .7540 by 60 to obtain 45.241 (ss.sss)

Dd°mm“ss.sss” = 33° & 28” & 45.241” = 33°28”45.241”

2.4.2 Special considerations for the use of Garmin® General Use Grade Receivers

The standard format for navigational purposes is decimal minutes (dd°mm.mmm”). This format is utilized due to the fact that nautical navigation charts are set up in this format. However, location information must be converted to a decimal degree (dd.ddddd°) format in order for GIS software to properly interpret the information. The conversion is accomplished by dividing the minutes portion of the coordinates by 60.

Converting to decimal degrees from decimal minutes:

$dd.ddddd^{\circ} = dd + (mm.mmm/60)$

Example: Convert 81°49.386 degrees to decimal degrees

$81 + (49.386/60) = 81.8231$ degrees

The reverse conversion is accomplished as follows:

$dd^{\circ}mm.mmm'' = dd \text{ \& } (.dddd*60)$

Example: Convert 81.8231 degrees to decimal minutes (dd°mm.mmm”)

Multiply .8231 by 60 to obtain 49.386 (mm.mmm)

$81^{\circ} \text{ \& } 49.386'' = 81^{\circ}49.386''$

GPS users need to familiarize themselves with the differences between the two formats, as they appear similar. Spreadsheets can automate the conversion process.

2.5 Records

The GPS coordinates and the SESD equipment identification number of the GPS receiver should be recorded in field logbooks at the time of GPS coordinate collection. The data logging capability of receivers may be used in lieu of the requirement to record the coordinates in logbooks when the following conditions can be met:

1. The location can easily be found later if it needs to be resurveyed prior to demobilization. A permanent monitoring well can easily be resurveyed, while most open-water work would not afford this opportunity.
2. The data is downloaded and ascertained to meet the accuracy requirements for the project prior to demobilization from the site.
3. The data is stored in at least two separate locations for transport, such as a laptop hard drive and a flash drive or compact disc.

In all cases where positions are electronically recorded, the provisions of the Electronic Records section of the SESD Operating Procedure for Control of Records (SESDPROC-002) should be followed.

Where locational data is collected and processed electronically, but not reported explicitly in the final report, a copy of the coordinates in text format should be output and entered into the project file in paper or electronic form. The output should include:

1. Latitude, generally in dd.ddddddd format.
2. Longitude, generally in dd.ddddddd format.
3. Date of collection.
4. DOP information where it supports the accuracy requirements.
5. Correction status of each point where it supports the accuracy requirements.
6. The datum used for the export.

Trimble® Pathfinder Office will create files with this information by exporting to a text file. The information will be contained in the .pos and .inf files.

Each equipment log will contain an equipment sign-out/sign-in form. Equipment defects or damage that is identified while in the field should be noted on the FEC Load-In Form (SESD FORM-011) when the equipment is signed in.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **In Situ Water Quality Monitoring**

Effective Date: July 19, 2013

Number: SESDPROC-111-R3

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Revision History

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<p>SESDPROC-111-R3, <i>In Situ Water Quality Monitoring</i>, replaces SESDPROC-111-R2</p> <p>General: Corrected any typographical, grammatical, and/or editorial errors.</p> <p>Cover Page: Omitted John Deatrick as an author. Changed the Ecological Assessment Branch Chief from Bill Cosgrove to John Deatrick. Changed the FQM from Laura Ackerman to Bobby Lewis.</p> <p>Revision History: Changes were made to reflect the current practice of only including the most recent changes in the revision history.</p> <p>Section 1.2: Added the following statement – “Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.”</p> <p>Section 1.4.2: Replaced “instrument log book” with “Field Equipment Tracking System (FETS).”</p>	July 19, 2013
SESDPROC-111-R2, <i>In Situ Water Quality Monitoring</i> , replaces SESDPROC-111-R1	December 7, 2009
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SESDPROC-111-R0, <i>In Situ Water Quality Monitoring</i> , Original Issue	April 1, 2007

TABLE OF CONTENTS

1	General Information.....	4
1.1	Purpose.....	4
1.2	Scope/Application.....	4
1.3	Documentation/Verification	4
1.4	Precautions.....	4
1.4.1	<i>Safety</i>	4
1.4.2	<i>Equipment Handling</i>	5
1.4.3	<i>Calibration</i>	5
1.5	References	6
2	Methodology.....	7
2.1	General	7
2.2	Real-time Monitoring.....	7
2.3	Profiling.....	8
2.4	Unattended Deployment	9

Contents

1 General Information

1.1 Purpose

The purpose of this procedure is to document acceptable practices in the use of multiparameter data sondes in the monitoring of in situ water quality parameters and dye tracer.

1.2 Scope/Application

This procedure covers the use of multiparameter data sondes for monitoring of in situ water quality including real-time measurement, profiling, and unattended data logging. In situ water quality parameters may include dissolved oxygen (DO), temperature, pH, conductivity, turbidity and chlorophyll. This procedure also applies to use of data sondes for monitoring dye tracer. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 Precautions

1.4.1 Safety

Equipment must be handled in a safe manner. Safety issues related to calibration or measurement of a specific parameter are addressed in individual parameter procedures. In addition, safety precautions should be followed in the deployment of data sondes. For unattended deployment in wadeable systems, data sondes should only be deployed and retrieved under safe flow/stage conditions. When deploying from a bridge, an amber flashing light should be operated on the roof of the field vehicle. When deploying from a boat, standard boating safety procedures should be followed. The SESD Safety, Health and Environmental Management Program Procedures and Policy Manual, provides more information regarding field safety.

1.4.2 Equipment Handling

To ensure the safe and reliable operation of equipment, the manufacturers' directions for transport, cleaning, decontamination, storage and operation shall be followed. In general, upon return from the field and applicable data downloading, the batteries should be removed from the data sonde and the sonde washed via light brushing in warm, soapy water. Each probe should be cleaned and stored as directed by the manufacturer.

Prior to use, data sondes should be signed out in the Field Equipment Tracking System (FETS) according to SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). When unattended deployment is anticipated, pingers should be attached to the sonde, as feasible, to aid in recovery should the sonde be displaced during deployment.

1.4.3 Calibration

Prior to use, each sonde probe should be calibrated according to the specific parameter measurement procedure. However, because the sonde is a multi-probe unit, additional care must be taken to prevent cross-contamination of calibration standards. Similarly, calibration of multiple sonde units requires cross-contamination prevention procedures. Specifically, following immersion of the sonde probes into each calibration standard, all probes should be thoroughly rinsed in distilled or de-ionized water and the excess water shaken off or blotted dry with a lint-free wipe. Conductivity standards are much more sensitive to cross contamination/dilution than other standards; therefore, prior to immersion in a conductivity standard, all probes should be thoroughly rinsed and completely dried with lint-free wipes or compressed air. The conductivity probe on the sondes provides a linear reading of conductivity across the scale, so it is no longer necessary, as in some older technology meters, to calibrate with a standard close to what one may expect in the field. Therefore, due to the propensity of the standard to be easily diluted, one should use a relatively high concentration standard (typically in the 10,000 umho range) for conductivity calibrations.

Besides being easily diluted, conductivity also affects other parameters (specifically DO), therefore conductivity should always be the first parameter calibrated. The recommended order for calibration of the individual probes on a multiparameter sonde is as follows:

1. Conductivity
2. pH
3. DO
4. Turbidity/Chlorophyll/Rhodamine in any order

Rhodamine and Chlorophyll probes are calibrated in a similar fashion to turbidity. Specifically, the zero level is set using DI or distilled water followed by calibration to a known standard (typically 100 ppb for Rhodamine).

1.5 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Field Measurement of Dissolved Oxygen, SESDPROC-106, Most Recent Version

SESD Operating Procedure for Field pH Measurement, SESDPROC-100, Most Recent Version

SESD Operating Procedure for Field Specific Conductance Measurement, SESDPROC-101, Most Recent Version

SESD Operating Procedure for Field Turbidity Measurement, SESDPROC-103, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

2 Methodology

2.1 General

With multiple probe options and customizable configuration, data sondes are extremely versatile tools for the measurement of in situ water quality. Effective use of multi-parameter sondes takes additional planning and procedures beyond those described in the individual operating procedures for each parameter (i.e., DO measurement, pH measurement, etc.).

Data sondes may be operated and/or programmed via the manufacturer's display unit or a laptop computer. In either case, it is recommended that the user take the manufacturer's applicable User Manual in the field should difficulties be encountered. If the display unit does not have a power indicator, the batteries should be checked or the unit charged, as applicable, prior to use. Power to the sonde may be supplied by the display unit or by the internal batteries installed in the sonde (a setting on the display unit menu). If the sonde is being powered by the display unit, it is possible to calibrate and set up the sonde for unattended deployment, when in fact there are no batteries in the sonde (the battery voltage being read is for the display unit and not for the sonde). Therefore, it is very important to insure that there are actually batteries in the sonde. Calibration and setting up for an unattended deployment use very little battery power, therefore, it is recommended that sondes be powered from their own internal batteries and not from the display unit. New alkaline or freshly charged nickel metal hydride (NMIH) batteries should be installed in each sonde prior to each field study. Generally, if the sondes will be deployed on multiple occasions during a field study, new alkaline batteries should be installed when the sonde voltage falls below 11.5 volts at end check. Nickel metal hydride (NMIH) batteries operate at a lower voltage than alkaline (1.2 volts vs. 1.5 volts); therefore, if using rechargeable batteries, they should be recharged or replaced if voltage falls below 10.5 volts.

Specific units require that, for the parameters of interest, the appropriate sensor be enabled via the display or laptop prior to use. The field investigator should follow manufacturer's procedures to ensure all required probes are functioning. If a particular parameter is not needed, the sensor should be turned off, via the menu, in order to conserve battery power. It should be noted that turning the reporting of the parameter off does not turn off the probe, it simply turns off the display of the parameter (the parameter is still being logged). One must go into the "Sensor" menu to actually turn off the sensor.

2.2 Real-time Monitoring

Real-time monitoring entails observing monitoring data via display unit or laptop computer as data is collected by the sonde. This data may be recorded in a field log book or logged to the internal memory of the sonde if so equipped. Logged data should be downloaded to a laptop or desktop computer as soon as possible. It is also recommended

that download files be backed up in a separate location (USB thumb drives work very well for this). In addition, even when logging data at regular intervals, it is recommended for real-time monitoring that data also be recorded in a field log book at some, likely less frequent, interval to ensure that some data is captured should the instrument logger fail.

Real-time monitoring generally involves hand-held deployment or attachment to a stationary object at the monitoring location. Hand-held deployments are useful for short-term monitoring in small, wadeable streams. For longer monitoring periods or to hold the sonde at a specific depth, attachment to a fixed object may be more effective. Fixed objects may include rocks or embedded logs already in place at the site or may include fence posts or rods placed by the field investigator prior to monitoring. Sondes may also be hung at desired depths from a boat on larger water bodies.

2.3 Profiling

Profiling involves real-time monitoring or individual measurements at several depths through a water column. Profiling is especially useful for documenting water column gradients or stratification of in situ parameters or for evaluating complete mix conditions in dye tracer studies. Profiling deployments are generally conducted by hand to provide the movement of the sonde through the water column; however, profiling can also be conducted using mechanical/ electrical winch or reel type devices. In profiling applications, the profiling cable should be labeled in some manner to indicate depth or the sonde calibrated for depth. In general, profiling data is recorded in a field log book along with the location and depth information for each measurement.

In fast moving waters it may be necessary to attach weight to the sonde. Weights should always be attached to the probe guard or sonde body, not the individual probes. If attached to the probe guard, weights should be secured in such a way that the weights and attachments do not interfere with probe operation. In all real-time and profiling applications, especially when the sonde is weighted, it is important to ensure that the profiling cable is securely attached to the baling harness of the sonde to prevent a disconnection of the sonde and potential loss or damage to the sonde.

It is important to note that SESD has two general type of sondes, vented and non-vented. Each type of sonde has its own profiling cable. The difference is how the depth sensor works. Non-vented sondes have a standard pressure or depth sensor that can be zeroed out at the site and will then accurately measure depth, typically to within a half a foot or less. Vented sondes have a small hole in the center of the connector pins where the cable attaches and are typically used to accurately (+/- 0.01 feet) measure changes in water stage level in unattended deployments, but may also be used for profiling applications. In order for a vented sonde to accurately measure depth or stage, the sonde **MUST** be used with a vented cable which vents to the atmosphere. If a vented sonde is used with a vented cable, just zero the depth at the site and measure depth as with a non-vented sonde. If a vented sonde is used with a non-vented cable it will **NOT** give accurate depth.

readings. Also, since vented sondes are typically used for stage measurements, the depth sensor is only rated to a maximum depth of 30 feet, whereas non-vented sondes are typically rated to 200 feet.

2.4 Unattended Deployment

Unattended deployment entails pre-programming and deployment of a sonde at a specific location to log monitoring data in the absence of observation by a field investigator. Unattended deployments are useful for collecting data at regular intervals over extended monitoring periods, frequently up to 3 – 4 days. However, since no data are recorded by hand during the deployment, it is critical that the sonde be correctly programmed.

Programming of the sonde should follow the manufacturer's procedures for unattended deployment. The sonde may be programmed in the lab prior to a field study or programmed in the field. Programming of the sonde is typically accomplished either by the sonde's display unit or by laptop computer. Programming requires input of a start date/time, deployment duration, data log file name, and monitoring interval. Programming times should always be input in local time for the study area, unless otherwise noted in the field log. The field log book should also include the sonde identifier, the date/time of initial deployment, date/time of retrieval, deployment location, and sonde depth. Similarly, recorded times should be in local time for the study area.

In addition to enabling the required probes as described in Section 2.1, some units further require identification of the parameters to include in the logged data file. The field investigator should follow manufacturer's procedures to ensure all necessary data will be successfully logged.

U.S. Environmental Protection Agency
Region 4, Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Field Measurement of Oxidation-Reduction Potential (ORP)**


Effective Date: January 29, 2013

Number: SESDPROC-113-R1

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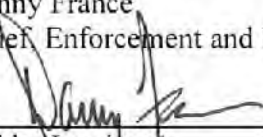
Signature: 

Date: 1-23-13

Approval

Name: Danny France

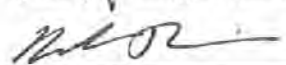
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Revision History

This Table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
<p>SESDPROC-113-R1, <i>Field Measurement of Oxidation-Reduction Potential (ORP)</i>, replaces SESDPROC-013-R0</p> <p>General: Corrected any typographical, grammatical, and/or editorial errors.</p> <p>Title Page: Changed the EIB Chief from Archie Lee to Danny France, and the Field Quality Manager from Laura Ackerman to Bobby Lewis.</p> <p>Section 1.2: Added the following statement: Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.</p> <p>Section 2.2: Added new paragraph #7 and new Figure 4. Renumbered subsequent Figures.</p> <p>In paragraph #8, deleted the following sentence: "In multi parameter sondes, the reference electrode is typically shared by the ORP and pH measuring systems."</p> <p>In paragraph #10, 2nd sentence, updated the reference from Figure 3 to Table 1. In addition, updated numbers in the "example" equation.</p> <p>In paragraph #11, 1st sentence, replaced +560 mV with +544 mV.</p> <p>Added new paragraph #12 and new Figure 6.</p> <p>Section 3.2: Added new paragraph #5 describing operational checks in the event of potential disturbances.</p> <p>In paragraph #8, added the following at the end of the 1st sentence: "or handled as directed by the SHEMP."</p>	January 29, 2013
SESDPROC-113-R0, <i>Field Measurement of Oxidation-Reduction Potential (ORP)</i> , Original Issue	August 7, 2009

TABLE OF CONTENTS

1	General Information	4
1.1	Purpose.....	4
1.2	Scope/Application	4
1.3	Documentation/Verification.....	4
1.4	References	4
1.5	General Considerations	5
1.5.1	<i>Safety</i>	5
1.5.2	<i>Records</i>	6
1.5.3	<i>Shipping</i>	6
2	Background	7
2.1	General.....	7
2.2	Instrumentation.....	7
2.3	Redox Chemistry	13
2.4	Applications	14
2.5	Limitations.....	15
3	Methodology	17
3.1	Standard Solutions.....	17
3.2	Verification and Calibration.....	17
3.3	Measurement	19
3.4	Reporting	19

1 General Information

1.1 Purpose

This document describes procedures, methods and considerations to be used and observed when conducting field Oxidation-Reduction Potential (ORP) measurements in aqueous environmental media, including groundwater, surface water and certain wastewater. The measurement of soil ORP is a non-standard measurement and procedures should be developed on a project-specific basis.

1.2 Scope/Application

This document describes procedures generic to all ORP measurement methods to be used by Science and Ecosystem Support Division (SESD) field personnel when collecting and handling samples in the field. On the occasion SESD personnel determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to obtain an ORP measurement, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the SESD LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

Faulkner, S.P., W.H. Patrick, Jr., and R.P. Gambrell. 1989. Field techniques for measuring wetland soil parameters. *Soil Sci. Soc. Am. J.* 53:883-890.

Megonigal, J.P., W.H. Patrick, Jr., and S.P. Faulkner. 1993. Wetland identification in seasonally flooded forest soils: soil morphology and redox dynamics. *Soil Sci. Soc. Am. J.* 57:140-149.

D.K. Nordstrom and F.D. Wilde. 2005. National Field Manual, Chapter A6, Section 6.5: Reduction Oxidation Potential (Electrode Method). USGS.

Pankow, J.E. 1991. Aquatic chemistry concepts. Lewis Publishers, Inc. Chelsea, Michigan. USA.

Pruitt, B.A. 2001. Hydrologic and soil conditions across hydrogeomorphic settings. Dissertation. The University of Georgia, Athens, GA. USA.

Soil Survey Staff. 1998. Keys to soil taxonomy, 8th Edition. United States Department of Agriculture, Natural Resources Conservation Service, Washington, DC. USA.

Standard Methods. 1992. Standard Methods for the Examination of Water and Wastewater, 18th Edition. Prepared and published jointly by: American Public Health Association, American Water Works Association, Water Environment Federation. American Public Health Association, Washington, DC. USA.

Stumm, W. and J.J. Morgan. 1981. Aquatic chemistry: an introduction emphasizing chemical equilibria in natural waters, 2nd Ed. John Wiley & Sons, New York. USA.

USEPA. 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division, Athens, GA.

USEPA. 2007. Safety, Health and Environmental Management Program Procedures and Policy Manual. Science and Ecosystem Support Division, Region 4, Athens, GA.

Wikipedia entry. Reduction Potential. http://en.wikipedia.org/wiki/Reduction_potential. Retrieved April 2, 2009.

1.5 General Considerations

1.5.1 Safety

Proper safety precautions must be observed when verifying or calibrating instruments for measurement of Oxidation-Reduction Potential. Refer to the SESD Safety, Health and Environmental Management Program Procedures and Policy Manual (most recent version) and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines should be used to complement the judgment of an experienced professional.

Reagents commonly used in the preparation of ORP calibration standards are toxic and require care when handling. When using this procedure, avoid exposure to these materials through the use of protective clothing, eye wear and gloves. Safety precautions when handling and preparing verification solutions should include gloves and eyewear to prevent dermal and eye contact, and a mask to avoid inhaling dust particles when handling dry materials. Vigorous flushing should be used if the reagents or solutions come in contact with skin or eyes. Following is specific information on commonly used solutions. The application of the solutions is described in detail in Section 3.1, Standard Solutions, of this procedure.

- Quinhydrone (CAS# 106-34-3) is a skin and respiratory irritant and is poisonous if ingested. Safety precautions when handling quinhydrone should include gloves to prevent dermal contact and a mask to avoid inhaling dust particles when mixing dry material to prepare calibration standards. Vigorous flushing should be used if

- concentrated material comes in contact with skin or eyes.
- Zobell's solution is also an irritant and toxic if ingested. The same handling precautions apply when mixing and using Zobell's solution as when using quinhydrone. Zobell's reacts with acid to form harmful byproducts, including hydrocyanide gas.
- Light's solution contains ferro- and ferric-cyanide compounds in sulfuric acid. The components are toxic and burns are possible from contact with this solution.
- Potassium iodide solutions have lower toxicity than most calibration solution options. General ingestion, skin contact, and eye contact precautions apply.

Unused quinhydrone, Zobell's, Light's or other calibration reagents and solutions should be returned to SESD for disposal in accordance with the SESD Safety, Health, and Environmental Management Plan (SHEMP).

1.5.2 Records

Documentation of field activities is done in a bound logbook. All records, including a unique, traceable identifier for the instrument, should be entered according to the procedures outlined in the SESD Operating Procedure for Logbooks (SESDPROC-010, most recent version) and the SESD Operating Procedure for Equipment Inventory and Management, (SESDPROC-108, most recent version).

All field ORP measurements pertinent to the sampling event should be recorded in the field logbook for the event as outlined in the SESD Operating Procedure for Logbooks (SESDPROC-010, most recent version), or managed electronically with appropriate backups as described in SESD Operating Procedure for Control of Records (SESDPROC-002, most recent version).

1.5.3 Shipping

Shipped material shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.

All shipping documents, such as bills of lading, will be retained by the project leader and stored in a secure place.

2 Background

2.1 General

Oxidation is the process of liberating electrons or gaining oxygen. Examples of oxidation include conversion of elemental iron to rust, elemental sulfur to sulfate, and elemental hydrogen to water (Pankow 1991). Reduction is the process of gaining electrons resulting in the charge on some atomic unit in the species to be reduced. Oxidation-reduction potential (ORP) or redox potential (hereafter, referred to as redox) is a measure of the intensity or activity of an aqueous environment or soil to mediate reactions of important elements in biological systems (e.g., O, N, Mn, Fe, S, and C) and other metallic elements.

Considerable confusion arises on the use of the terms oxidation and reduction as they apply to the media under study. The following introduction reproduced from an online 'Wikipedia' article on the topic lucidly explains their relationship in ORP measurement:

Reduction potential (also known as **redox potential**, **oxidation / reduction potential** or **ORP**) is the tendency of a chemical species to acquire electrons and thereby be reduced. Each species has its own intrinsic reduction potential; the more positive the potential, the greater the species' affinity for electrons and tendency to be reduced.

In aqueous solutions, the reduction potential is the tendency of the solution to either gain or lose electrons when it is subject to change by introduction of a new species. A solution with a higher (more positive) reduction potential than the new species will have a tendency to gain electrons from the new species (i.e. to be reduced by oxidizing the new species) and a solution with a lower (more negative) reduction potential will have a tendency to lose electrons to the new species (i.e. to be oxidized by reducing the new species). Just as the transfer of hydrogen ions between chemical species determines the pH of an aqueous solution, the transfer of electrons between chemical species determines the reduction potential of an aqueous solution. Like pH, the reduction potential represents an intensity factor. It does not characterize the capacity of the system for oxidation or reduction, in much the same way that pH does not characterize the buffering capacity.

In short, a numerically positive redox potential or ORP represents an environment conducive to the oxidation of an introduced substance by reduction of the original media.

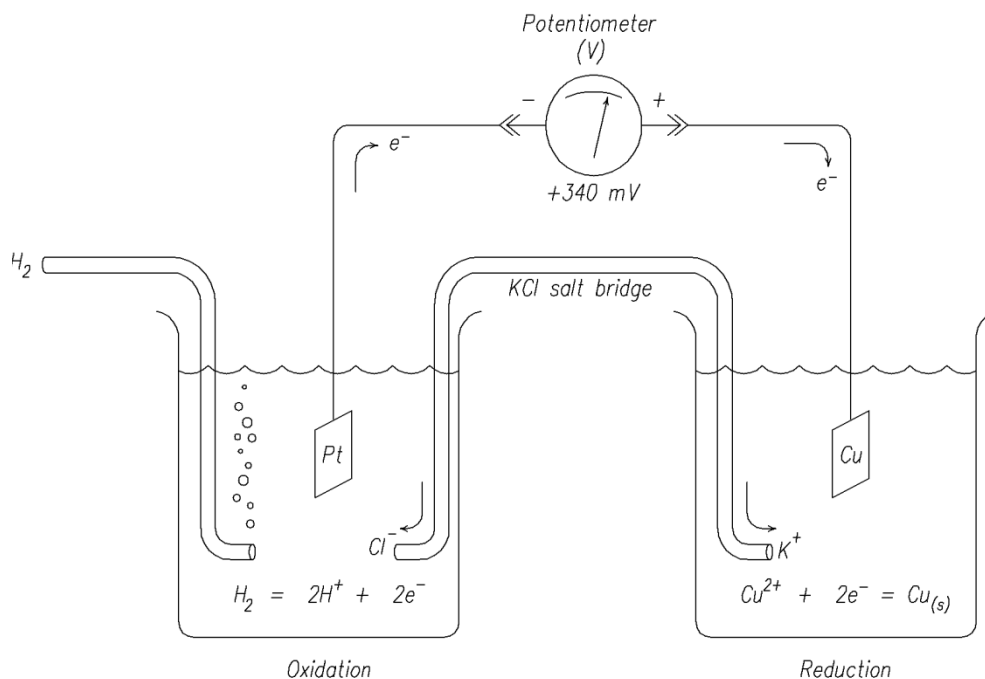
2.2 Instrumentation

ORP measurement systems are a practical implementation of electrochemical cells, which use metal electrodes in a solution to generate an electric current or voltage. If a platinum electrode is immersed in water with hydrogen bubbled into the solution, the H^2 is oxidized as follows:



In the electrochemical half-cell illustrated below in Fig.1, hydrogen gas oxidizes to hydrogen ions and free electrons, comprising an oxidation-reduction couple. This couple reaches an

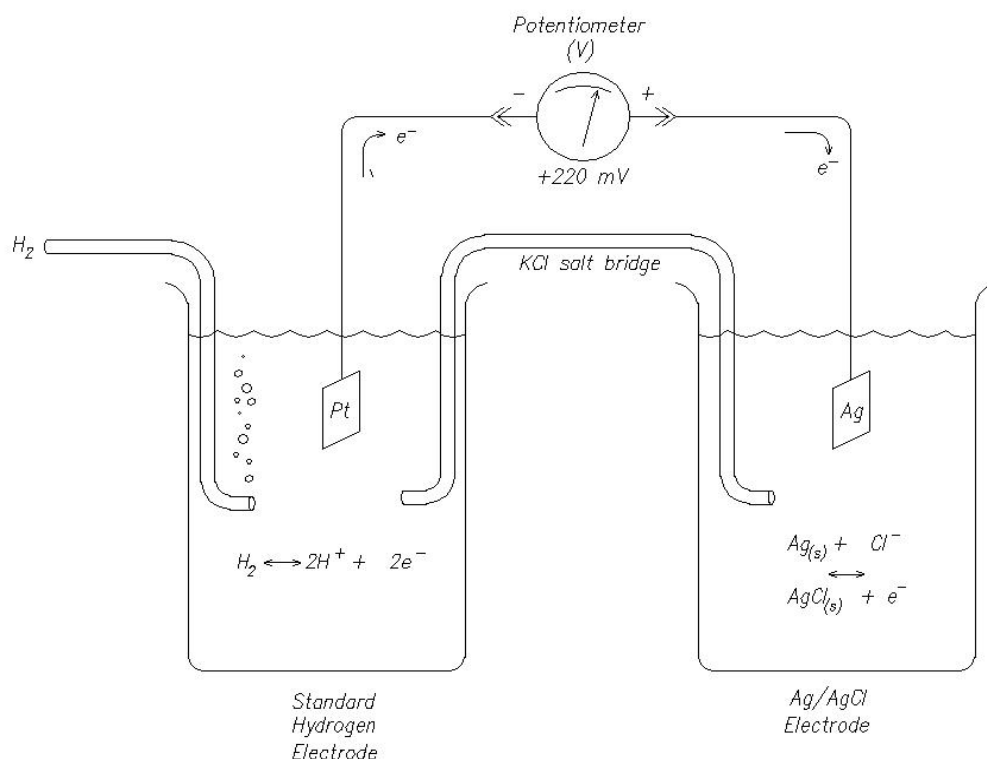
Figure 2



In field practice, the hydrogen electrode is difficult to reproduce. To conduct field measurements, a reference electrode is needed that is simple to maintain and will generate a potential that can be referenced to the standard hydrogen electrode. These requirements are met by the Saturated Calomel Electrode (SCE) and the Silver/Silver Chloride Electrode (SSCE - the SSCE is also commonly identified as an Ag/AgCl electrode). The SCE contains a small amount of elemental mercury, and while useful for certain applications, would rarely be used at SESD. The SSCE or Ag/AgCl electrode is generally used as the reference cell in SESD instrumentation.

In Figure 3 below, a SHE is connected to an Ag/AgCl electrode. In this example of an electrochemical cell, both cells reach an equilibrium potential. At that equilibrium state, the potential of the Ag/AgCl cell is 220mV more positive than the standard hydrogen electrode.

Figure 3



This half-cell potential of the Ag/AgCl electrode in reference to the SHE is used to convert measurements taken with an Ag/AgCl reference back to the hydrogen scale. While the laboratory Ag/AgCl half-cell shown has a potential of +220mV, practical reference cells have varying potentials based on temperature and filling solutions as shown in Table 1 below.

Table 1

Half-cell Potential of Ag/AgCl reference electrode

derived from USGS NFM, Table 6.5.2 (9/2005)

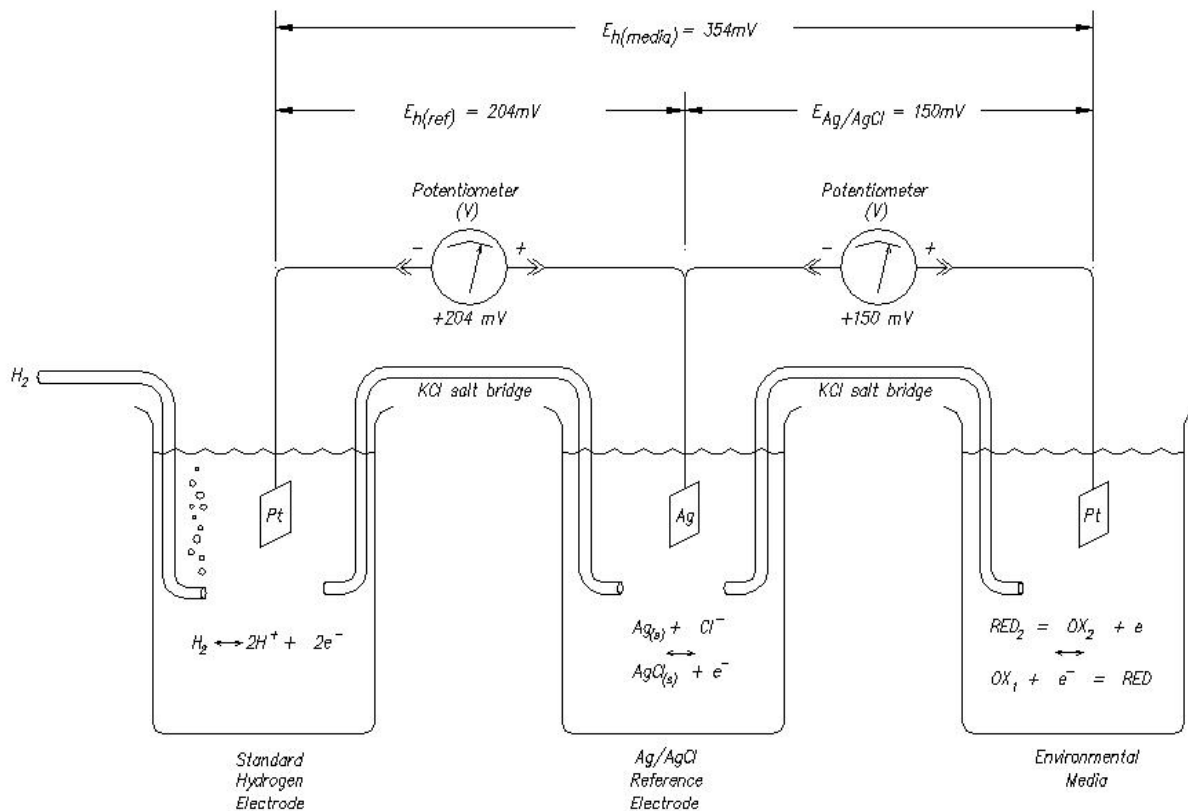
T(°C)	Molarity of KCl filling solution			
	3M	3.3M*	3.5M	Sat/4M
10	220	217	215	214
15	216	214	212	209
20	213	210	208	204
25	209	207	205	199
30	205	203	201	194
35	202	199	197	189
40	198	195	193	184

*interpolated value

Note: YSI sondes and Thermo electrodes typically use 4M KCl filling solutions. Eureka sondes typically use 3.3M KCl filling solutions.

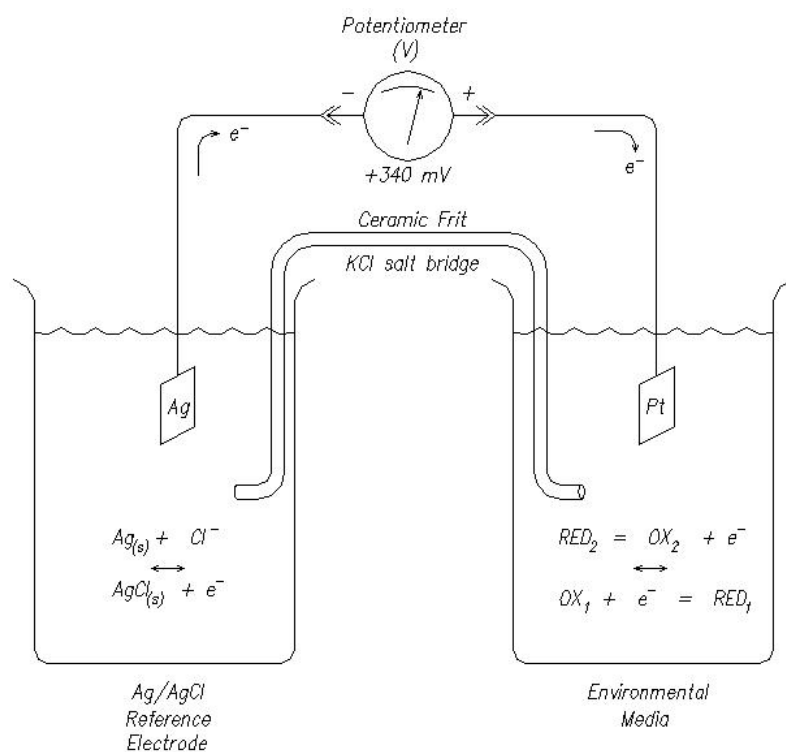
In Figure 4, below, the relationship between a hydrogen electrode, a reference electrode, and a platinum sensing electrode in an arbitrary media is shown. In this case, the ORP of the media in reference to the silver/silver chloride electrode is 150mV. To obtain Eh, the potential of the reference electrode in relation to a hydrogen electrode is added to the potential of the sensing electrode in relation to the reference electrode. In practice, the potential of the reference electrode in relation to a hydrogen electrode is not measured, but obtained from Table 1 above.

Figure 4



In Figure 5 below, a field instrument is represented as separate electrochemical cells. The Ag/AgCl reference electrode uses a ceramic frit or other means to provide the essential salt bridge to the environmental media. The platinum sensing electrode is immersed in the environmental media and connected internally in the instrument to measure the potential (voltage) between the two electrodes.

Figure 5



In this illustration, the ORP is measured as 340 mV. This measurement is made in reference to the Ag/AgCl reference electrode and would be reported as such, or as $E_{Ag/AgCl} = 340\text{mV}$.

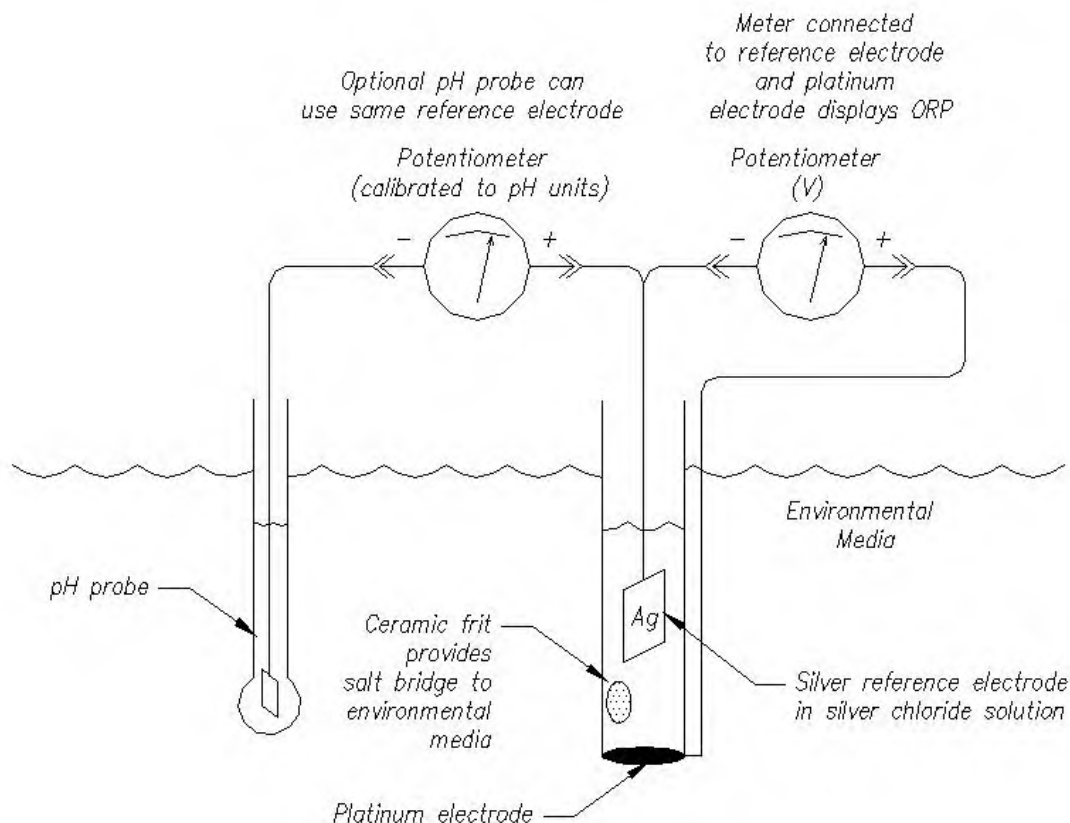
In some cases it will be desirable to report the reading on the hydrogen scale, or Eh. To do so, the potential of the reference electrode against the SHE, obtained from Table 1, is added to $E_{Ag/AgCl}$. For our example:

$$\begin{array}{rcl}
 340 \text{ mV} & \text{Measured ORP (} E_{Ag/AgCl} \text{) of sample} & \\
 + \quad \underline{204 \text{ mV}} & \text{Eh of Ag/AgCl electrode (ORP of Ag/AgCl electrode referenced to SHE)} & \\
 544 \text{ mV} & \text{Eh of sample} &
 \end{array}$$

Both the +340 mV field reading and the corrected +544 mV Eh can properly be referred to as ORP results. It is only through specifying the reference scale that the ambiguity can be eliminated.

In Figure 6, below, the theoretical cells shown above have been configured as a practical field instrument. The salt bridge is commonly provided by a ceramic frit connecting the environmental media to the reference electrode. In multi-parameter sondes, the pH probe commonly uses the same reference electrode as the ORP probe.

Figure 6



2.3 Redox Chemistry

In acid-base chemistry, the pH of a system is defined as the negative logarithm of the hydrogen ion activity (simplified in practice to the hydrogen ion concentration):

$$\text{pH} = -\log \{ \text{H}^+ \}$$

Similarly, Pankow (1991) described the negative logarithm of the electron activity (pe) as the master variable for describing the equilibrium position for all redox couples in a given system:

$$\text{pe} / -\log \{ \text{e}^- \}$$

It can be shown (Pankow) that pe is related to Eh by

$$\text{Eh} = \text{pe} * (2.303 * \text{R} * \text{T}) / \text{F}$$

Where:

$$\begin{aligned} R &= \text{gas constant} = 8.314 \text{ J K}^{-1} \text{ mol}^{-1} \\ T &= \text{temperature, } ^\circ\text{K} \\ F &= \text{Faraday constant} = 96.485 \times 10^3 \text{ C mol}^{-1} \end{aligned}$$

At 25°C (298°K) this simplifies to

$$E_H = pe * 0.05916$$

And

$$pe = E_H / 0.05916$$

According to Faulkner et al. (1989) redox is a quantitative measure of electron availability and is indicative of the intensity of oxidation or reduction in both chemical and biological systems. When based on a hydrogen scale, redox (E_H) is derived from the Nernst Equation (Stumm and Morgan 1981):

$$E_H = E_H^{\circ} + \frac{2.303}{n} \frac{RT}{F} \log \left(\frac{J_{\text{ox}}^{n_i}}{J_{\text{red}}^{n_j}} \right)$$

Where:

$$\begin{aligned} E_H^{\circ} &= \text{potential of reference, mV} \\ R &= \text{gas constant} = 81.987 \text{ cal deg}^{-1} \text{ mole}^{-1} \\ T &= \text{temperature, } ^\circ\text{K} \\ n &= \text{number of moles of electrons transferred} \\ F &= \text{Faraday constant} = 23.061 \text{ cal/mole-mv} \\ \{\text{ox}\} \text{ and } \{\text{red}\} &= \text{activity of the oxidants and reductants, respectively} \end{aligned}$$

2.4 Applications

When interpreted properly, redox combined with other conventional water quality parameters is useful in developing a more complete understanding of water chemistry. Several applications of redox are identified below:

1. Redox could be viewed as an extension of the oxygen scale. In this model, the DO probe spans the aerobic scale and the redox probe extends that scale to measure anaerobic conditions. Inferences to geochemistry and chemical speciation can be made from the oxidative state of the system. Application to metal sequestration, metal-iron, -sulfide, -methane complexation, and the subsequent bioaccumulation potential is possible.
2. Redox can be used to identify anaerobiosis at or near the water column and sediment interface in streams, lakes, and estuaries.
3. Redox may be useful in determination of stream jurisdiction and wetland delineation in that it can indicate conditions of soil saturation.

4. Based on redox, a pe (or EH) vs. pH stability diagram can be developed to aid in nutrient exchange studies including the timing, release, and partitioning of important water and sediment quality pollutants such as nitrogen and phosphorus species. Most importantly, redox can be used to address error associated with chamber-effect during closed chamber measurements of the water-sediment interface. Redox probes placed inside the contact chamber and inserted approximately ten centimeters into the underlying sediment can be used to monitor changes in sediment redox caused by the chamber, and steps can be taken to reduce chamber-effect.
5. Redox may be useful in establishing water and sediment quality standards applicable to wetlands.
6. Redox is used to assess the potential of a groundwater system to support various in situ reactions with contaminants, such as reductive dechlorination of chlorinated solvents.
7. Redox can provide a useful indicator of conditions that might compromise the performance of Clark-type dissolved oxygen (DO) probes. In general, anaerobic conditions occur at a redox range of +150 mV to +300 mV (pH-dependent and adjusted to hydrogen reference electrode). When redox drops below this level, DO measurements as determined with a Clarke-type probe are highly suspect as the semi-permeable membrane does not discriminate between partial O₂ and sulfides. Consequently, the meter may be reading sulfides.

2.5 Limitations

In most environmental media, redox reactions will not reach equilibrium due to low concentrations or multiple redox species. Consequently, redox measurements can generally be considered semi-quantitative in environmental media, unless certain conditions exist.

The USGS in the Interferences and Limitations Section 6.5.3A of their National Field Manual succinctly describe some of the issues encountered in the application of ORP measurements. This section is reproduced here, unedited:

6.5.3.A INTERFERENCES AND LIMITATIONS

Measurements should not be carried out without an awareness of the interferences and limitations inherent in the method.

- *Organic matter and sulfide may cause contamination of the electrode surface, salt bridge, or internal electrolyte, which can cause drift or erratic performance when reference electrodes are used (American Public Health Association and others, 2001).*
- *Hydrogen sulfide can produce a coating on the platinum electrode that interferes with the measurement if the electrode is left in sulfide-rich water for several hours (Whitfield, 1974; Sato, 1960).*
- *The platinum single and combination redox electrodes may yield unstable readings in solutions containing chromium, uranium, vanadium, or titanium ions and other ions that*

are stronger reducing agents than hydrogen or platinum (Orion Research Instruction Manual, written commun., 1991).

- *Do not insert redox electrodes into iron-rich waters directly after the electrode(s) contact ZoBell's. An insoluble blue precipitate coats the electrode surface because of an immediate reaction between ferro- and ferricyanide ions in ZoBell's with ferrous and ferric ions in the sample water, causing erratic readings.*

Many elements with more than one oxidation state do not exhibit reversible behavior at the platinum electrode surface and some systems will give mixed potentials, depending on the presence of several different couples (Barcelona and others, 1989; Bricker, 1982, p. 59–65; Stumm and Morgan, 1981, p. 490–495; Bricker, 1965, p. 65). Methane, bicarbonate, nitrogen gas, sulfate, and dissolved oxygen generally are not in equilibrium with platinum electrodes (Berner, 1981).

TECHNICAL NOTE:

Misconceptions regarding the analogy between Eh (pe) and pH as master variables and limitations on the interpretation of Eh measurements are explained in Hostettler (1984), Lindberg and Runnells (1984), Thorstenson (1984), and Berner (1981). To summarize:

(1) Hydrated electrons do not exist in meaningful concentrations in most aqueous systems—in contrast, pH represents real activities of hydrated protons. Eh may be expressed as pe (the negative logarithm of the electron activity), but conversion to pe offers no advantage when dealing with measured potentials.

(2) Do not assume that redox species coexist in equilibrium. Many situations have been documented in which dissolved oxygen coexists with hydrogen sulfide, methane, and ferrous iron.

- *The practicality of Eh measurements is limited to iron in acidic mine waters and sulfide in waters undergoing sulfate reduction.*
- *Other redox species are not sufficiently electroactive to establish an equilibrium potential at the surface of the conducting electrode.*

(3) A single redox potential cannot be assigned to a disequilibrium system, nor can it be assigned to a water sample without specifying the particular redox species to which it refers. Different redox elements (iron, manganese, sulfur, selenium, arsenic) tend not to reach overall equilibrium in most natural water systems; therefore, a single Eh measurement generally does not represent the system.

3 Methodology

3.1 Standard Solutions

Care should be taken not to contaminate standards and samples and to verify the expiration date of all standards prior to use. All meters should be verified or calibrated according to the manufacturer's procedures.

Standard solutions for calibration and verification should be selected to meet project requirements. SESD generally maintains a stock of Zobell's solution suitable for most projects. The characteristics and use of the common standard solutions are described below.

- Zobell's solution contains potassium ferri- and ferro- cyanide compounds. The solution is available as prepared solutions or premeasured reagents for mixing by the user. Zobell's has moderate toxicity but will react with acid to form harmful byproducts, including hydrocyanide gas. It has a shelf life ranging from several days to several months depending on the manufacturer. Stock and working solutions of Zobell's should be stored in dark bottles due to its light sensitivity.
- Quinhydrone solutions are mixed at the time of use by adding quinhydrone to pH 4 or pH 7 buffers. At 25°C, the E_h of quinhydrone pH 4 and pH 7 verification solutions are 462mV and 285mV respectively. An advantage of quinhydrone solutions is that they offer a span of calibration points that may be appropriate for particular applications. Quinhydrone is a lightly 'poised' solution in that it offers less driving force towards the calibration point: a compromised instrument is more likely to be revealed in a quinhydrone calibration. A quinhydrone calibration/verification solution is created by adding 10g of quinhydrone to 1L of pH 4 or pH 7 buffer solution (ASTM D1498). The solutions are mixed on a magnetic mixing plate for a minimum of 15 minutes to create a saturated solution with undissolved crystals remaining. Quinhydrone solutions are usable for 8 hrs from the time of mixing.
- Light's solution consists of ferrous and ferric ammonium sulphate in sulphuric acid. The solution would rarely be used at SESD due to its high acidity and associated handling difficulty. Spent solutions with a pH<2 would be regulated as a hazardous waste. Light's is a highly poised solution that may allow a marginally functioning electrode to pass calibration.
- A prepared potassium iodide solution is available which has low toxicity and a long shelf life. The solution may stain clothing or surfaces if spilled.

3.2 Verification and Calibration

ORP instruments may be **verified** or **calibrated**, depending on the application. The approach chosen should be selected based on project needs and information presented in Section 2.4., Limitations. Standard laboratory practice in making ORP measurements is to **verify** the accuracy of the instrument prior to use, and this practice should be followed when true quantitative results

are required. In a **verification**, the instrument in its direct-reading mode is checked against a standard solution in a pass/no-pass test, and no corrections are applied to subsequent measurements. In most applications, the ORP information is used semi-quantitatively and for these applications, the instruments may be **calibrated** to the standard solutions. In an instrument **calibration**, the instrument probe is placed in the standard solution and the difference between the standard measurement and the known ORP value of the standard is used by the instrument to make adjustments to the subsequent measurements.

In **verification** of an ORP instrument, the instrument is set to absolute mV reading mode or the internal calibration offset is zeroed out. The instrument probe should then be placed in the standard solution and the reading verified to fall within ± 10 mV of the predicted reading for the standard. Instruments with single-purpose electrodes are most suitable for this approach. If the instrument fails the verification, standard solution quality should be considered and instrument maintenance performed per the manufacturer's procedures.

In most SESD field practice, the end data use is semi-quantitative. In this case, the instruments can be **calibrated** to standard solutions appropriate for the project using the manufacturer's recommended procedure. One minute after the calibration, the instrument should display a stable reading within ± 10 mV of the predicted reading. An instrument failing this test should be recalibrated to determine if the problem is inadequate equilibration time. In the event of continued instrument failure, aging or contamination of the standard solution should be considered. Subsequently the electrode should be serviced according to the manufacturer's procedures. Common service procedures include cleaning the platinum electrode with mild abrasives or acids and refilling or replacing the reference electrode.

Prior to a mobilization, all ORP instruments will be checked for proper operation and verified or calibrated against standard solutions. During the field mobilization, each instrument will be calibrated or verified prior to, and verified after, each day's use or deployment.

Even though it is not necessary to re-calibrate ORP instrument at regular intervals during the day, it may be appropriate to occasionally perform operational checks to determine if site conditions, such as an extreme temperature change or submersion of a filling solution port have impacted the instrument's performance. If an operational check is warranted, the field operator should follow the appropriate verification/calibration steps as described above.

The predicted ORP values of standard solutions will be obtained from the manufacturer of prepared solutions, literature, or appropriate values listed in this procedure. Care is in order, as the predicted ORP value is specific for the type of reference electrode used by the probe (either Ag/AgCl or calomel) and the molarity of the filling solution in the reference electrode. To use the solution with another electrode or filling solution, the expected ORP readings for the solution should be converted to E_h for the probes intended for the solution as per the Reporting section of this procedure. Then a table can be compiled for the electrode in use by subtracting the $E_{h,ref}$ for the electrode and filling solution in use. This will be done at the Field Equipment Center (FEC) for the solutions stocked.

Verification solutions should be managed per the manufacturer's directions regarding storage and handling. After instrument verification or calibration, the solution cannot be returned to the stock solution container, although a separate container of working solution can be maintained.

Spent solutions and working solutions should be returned from the field to the SESD laboratory for proper disposal by the SHEMP, or handled as directed by the SHEMP. Properly handled stock solutions may be returned to the FEC for use at that facility.

3.3 Measurement

ORP measurements should be conducted in a fashion that prevents the addition or loss of any potential oxidants or reductants. Results could be compromised by exposing the sample to air or allowing H₂S to off-gas from anoxic samples. Like dissolved oxygen measurements, ORP measurements should be conducted in situ or by using a flow-through cell evacuated of air (see the SESD Operating Procedure for Field Measurement of Dissolved Oxygen (SESDPROC-106, most recent version).

When using multi-parameter probes for ORP measurements, the general guidelines for probe deployment described in the SESD Operating Procedure for Field Measurement of Dissolved Oxygen (SESDPROC-106, most recent version) and the SESD Operating Procedure for In situ Water Quality Monitoring (SESDPROC-111, most recent version) apply.

ORP probes must be operated and maintained in accordance with the manufacturer's instructions. Reference electrodes in multi-parameter probes may require regular filling or replacement. Single parameter ORP electrodes may require regular filling and operation in an upright position to assure that proper salt bridge flow is maintained. Platinum electrode surfaces are easily contaminated and polishing or cleaning of the electrodes should be performed as recommended by the manufacturer.

Measurements in field logbooks should be recorded to the nearest mV. The type of reference electrode in use and its filling solution should be recorded in at least one logbook as part of the field project records.

ORP is a temperature sensitive measurement, but ORP instruments are not temperature compensated. Consequently, the media temperature should always be recorded at the same time as the ORP is recorded. Likewise, as ORP is often pH dependent, pH should also be recorded at the time of ORP measurement.

3.4 Reporting

In the absence of a specified reference scale, ORP data has no meaning. Therefore, the reference scale used should always be specified in reporting or discussing the ORP data. ORP measurements converted to a hydrogen scale can be reported as "E_h". Data reported as the direct field measurement without correction might be described as "ORP referenced to Ag/AgCl electrode" or "E_{Ag/AgCl}". The expectations of the data user should be ascertained or the measurements should be reported in both systems.

Final reporting values of Eh or ORP should be rounded to the nearest 10mV. The following spreadsheet formula can perform the rounding of an interim result located in spreadsheet cell 'A1':

`=INT(A1/10+0.5)*10`

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

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Number: SESDPROC-202-R2

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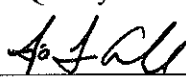
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<p>SESDPROC-202-R1, <i>Management of Investigation Derived Waste</i>, replaces SESDPROC-202-R0.</p> <p>General Corrected any typographical, grammatical and/or editorial errors.</p> <p>Title Page Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch.</p>	<p>November 1, 2007</p>

<p>Changed Bill Cosgrove's title from Acting Chief to Chief.</p> <p>Section 1.3 Updated information to reflect that the procedure is located on the H: drive of the LAN. Clarified Field Quality Manager (FQM) responsibilities.</p> <p>Section 1.4 Alphabetized and revised the referencing style for consistency.</p> <p>Section 1.5.1 Corrected the title of the Safety, Health, and Environmental Management Program Procedures and Policy Manual.</p>	
<p>SESDPROC-202-R0, Management of Investigation Derived Waste, Original Issue</p>	<p>February 05, 2007</p>

TABLE OF CONTENTS

1	General Information	5
1.1	Purpose	5
1.2	Scope/Application	5
1.3	Documentation/Verification	5
1.4	References.....	5
1.5	General Precautions	6
1.5.1	<i>Safety</i>	6
1.5.2	<i>Procedural Precautions</i>	6
2	Types of Investigation Derived Waste	7
3	Management of Non-Hazardous IDW.....	8
4	Management of Hazardous IDW	9

TABLES

Table 1:	Disposal of IDW.....	10
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Contents

1 General Information

1.1 Purpose

This document describes general and specific procedures and considerations to be used and observed when managing investigation derived waste (IDW) generated during the course of hazardous waste site investigations.

1.2 Scope/Application

The procedures and management options for the different categories of IDW described in this document are to be used by SESD field personnel to manage IDW generated during site investigations. On the occasion that SESD field personnel determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to manage IDW generated at a particular site, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

United States Environmental Protection Agency (US EPA). 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when managing IDW. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when managing IDW:

- Due to time limitations and restrictions posed by RCRA regulations on storage of hazardous waste, accumulation start dates should be identified on all drums, buckets or other containers used to hold IDW so that it can be managed in a timely manner.
- During generation of both non-hazardous and hazardous IDW, keep hazardous IDW segregated from non-hazardous IDW to minimize the volume of hazardous IDW that must be properly managed.

2 Types of Investigation Derived Waste

Materials which may become IDW include, but are not limited to:

- Personal protective equipment (PPE) - This includes disposable coveralls, gloves, booties, respirator canisters, splash suits, etc.
- Disposable equipment and items - This includes plastic ground and equipment covers, aluminum foil, conduit pipe, composite liquid waste samplers (COLIWASAs), Teflon® tubing, broken or unused sample containers, sample container boxes, tape, etc.
- Soil cuttings from drilling or hand augering.
- Drilling mud or water used for mud or water rotary drilling.
- Groundwater obtained through well development or well purging.
- Cleaning fluids such as spent solvents and wash water.
- Packing and shipping materials.

Table 1, found at the end of this procedure, lists the types of IDW commonly generated during field investigations and the current disposal practices for these materials.

For the purpose of determining the ultimate disposition of IDW, it is typically distinguished as being either hazardous or non-hazardous. This determination is based on either clear regulatory guidance or by subsequent analysis. This determination and subsequent management is the responsibility of the program site manager.

3 Management of Non-Hazardous IDW

Disposal of non-hazardous IDW should be addressed in the study plan or QAPP for the investigation. To reduce the volume of any IDW transported back to the Field Equipment Center (FEC), it may be necessary to compact the waste into a reusable container, such as a 55-gallon drum.

If the waste is from an active facility, permission should be sought from the operator of the facility to place the non-hazardous PPE, disposable equipment, and/or paper/cardboard into the facility's dumpsters. If necessary, these materials may be placed into municipal dumpsters, with the permission of the owner. These materials may also be taken to a nearby permitted landfill. On larger studies, waste hauling services may be obtained and a dumpster located at the study site.

Disposal of non-hazardous IDW such as drill cuttings, drilling mud, purge or development water, decontamination wash water, etc., should be specified in the approved study plan or QAPP. It is recommended that these materials be placed into a unit with an environmental permit, such as a landfill or sanitary sewer. These materials must not be placed into dumpsters. If the facility at which the study is being conducted is active, permission should be sought to place these types of IDW into the facility's treatment system. It may be feasible to spread drill cuttings around the borehole, or, if the well is temporary, to place the cuttings back into the borehole. Non-hazardous monitoring well purge or development water may also be poured onto the ground down gradient of the monitoring well when site conditions permit. Purge water from private potable wells which are in service may be discharged directly onto the ground surface.

The minimum requirements for this subsection are:

- Non-hazardous liquid and soil/sediment IDW may be placed on the ground or returned to the source if doing so does not endanger human health or the environment or violate federal or state regulations. Under no circumstances, however, should monitoring well purge water be placed back into the well from which it came.
- Soap and water decontamination fluids and rinsates of such cannot be placed in any water bodies and must be collected and returned to the FEC for disposition.
- The collection, handling and proposed disposal method must be specified in the approved study plan or QAPP.

4 Management of Hazardous IDW

Disposal of hazardous or suspected hazardous IDW must be specified in the approved study plan or QAPP for the study or investigation. Hazardous IDW must be disposed as specified in USEPA regulations. If appropriate, these wastes may be placed back in an active facility waste treatment system. These wastes may also be disposed in the source area from which they originated if doing so does not endanger human health or the environment.

If on-site disposal is not feasible, and if the wastes are suspected to be hazardous, appropriate tests must be conducted to make that determination. If they are determined to be hazardous wastes, they must be properly contained and labeled. They may be stored on the site for a maximum of 90 days before they must be manifested and shipped to a permitted treatment or disposal facility. Generation of hazardous IDW must be anticipated, if possible, to allow arrangements for proper containerization, labeling, transportation and disposal/treatment in accordance with USEPA regulations.

The generation of hazardous IDW should be minimized to conserve Division resources. Most routine studies should not produce any hazardous IDW, with the possible exception of spent solvents and, possibly, purged groundwater. The use of solvents during field cleaning of equipment should be minimized by using solvent-free cleaning procedures for routine cleaning and decontamination (see SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205). If solvents are needed, the volume should be minimized by using only the amount necessary and by capturing the residual solvent separately from the aqueous decontamination fluids (detergent/wash water mixes and water rinses).

At a minimum, the requirements of the management of hazardous IDW are as follows:

- Spent solvents must be left on-site with the permission of site operator and proper disposal arranged.
- All hazardous IDW must be containerized. Proper handling and disposal should be arranged prior to commencement of field activities.

Table 1: Disposal of IDW

TYPE	HAZARDOUS	NON - HAZARDOUS
PPE-Disposable	Containerize in plastic 5-gallon bucket with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise return to FEC for proper disposal.	Place waste in trash bag. Place in dumpster with permission of site operator, otherwise return to FEC for disposal in dumpster.
PPE-Reusable	Decontaminate as per SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, if possible. If the equipment cannot be decontaminated, containerize in plastic 5-gallon bucket with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise return to FEC for proper disposal.	Decontaminate as per SESDPROC-205, and return to FEC.
Spent Solvents	Containerize in original containers. Clearly identify contents. Leave on-site with permission of site operator and arrange for proper disposal.	N/A
Soil Cuttings	Containerize in DOT-approved container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in a 55-gallon steel drum with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal. **
Groundwater	Containerize in DOT-approved container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in an appropriate container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal. **
Decontamination Water	Containerize in DOT-approved container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in an appropriate container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal. Decontamination water may also be disposed in a sanitary sewer system, with permission from the wastewater treatment plant representative, and if doing so does not endanger human health or the environment, or violate federal or state regulations.
Disposable Equipment	Containerize in DOT-approved container or 5-gallon plastic bucket with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in an appropriate container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal. If unfeasible, return to FEC for disposal in dumpster.
Trash	N/A	Place waste in trash bag. Place in dumpster with permission of site operator, otherwise return to FEC for disposal in dumpster.

**** These materials may be placed on the ground if doing so does not endanger human health or the environment or violate federal or state regulations.**

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: Field Equipment Cleaning and Decontamination


Effective Date: December 20, 2011

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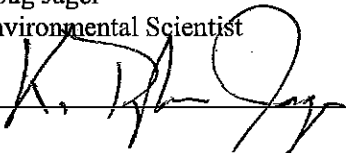
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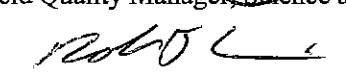
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<p>SESDPROC-205-R1, <i>Field Equipment Cleaning and Decontamination</i>, replaces SESDPROC-205-R0.</p> <p>General Corrected any typographical, grammatical and/or editorial errors.</p> <p>Title Page Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove's title from Acting Chief to Chief.</p> <p>Section 1.3 Updated information to reflect that the procedure is located on the H: drive of the LAN. Clarified Field Quality Manager (FQM) responsibilities.</p> <p>Section 1.5 Alphabetized and revised the referencing style for consistency.</p> <p>Section 1.6.1 Corrected the title of the Safety, Health, and Environmental Management Program Procedures and Policy Manual.</p>	November 1, 2007
<p>SESDPROC-205-R0, <i>Field Equipment Cleaning and Decontamination</i>, Original Issue</p>	February 05, 2007

TABLE OF CONTENTS

1	General Information.....	5
1.1	Purpose.....	5
1.2	Scope/Application	5
1.3	Documentation/Verification.....	5
1.4	Definitions.....	5
1.5	References.....	6
1.6	General Precautions.....	6
1.6.1	<i>Safety</i>	6
1.6.2	<i>Procedural Precaution</i>	7
2	Introduction to Field Equipment Cleaning and Decontamination	8
2.1	General.....	8
2.2	Handling Practices and Containers for Cleaning Solutions	8
2.4	Sample Collection Equipment Contaminated with Concentrated Materials	9
2.5	Sample Collection Equipment Contaminated with Environmental Media..	9
2.6	Handling of Decontaminated Equipment	10
3	Field Equipment Decontamination Procedures	11
3.1	General.....	11
3.2	Specifications for Decontamination Pads	11
3.3	"Classical Parameter" Sampling Equipment	12
3.4	Sampling Equipment used for the Collection of Trace Organic and Inorganic Compounds	12
3.5	Well Sounders or Tapes	13
3.6	Redi-Flo2® Pump	13
3.6.1	<i>Purge Only (Pump and Wetted Portion of Tubing or Hose)</i>	13
3.6.2	<i>Purge And Sample</i>	13
3.6.3	<i>Redi-Flo2® Ball Check Valve</i>	14
3.7	Downhole Drilling Equipment.....	15
3.7.1	<i>Introduction</i>	15
3.7.2	<i>Preliminary Cleaning and Inspection</i>	15
3.7.3	<i>Drill Rig Field Cleaning Procedure</i>	16
3.7.4	<i>Field Decontamination Procedure for Drilling Equipment</i>	16
3.7.5	<i>Field Decontamination Procedure for Direct Push Technology (DPT) Equipment</i>	17

1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when cleaning and decontaminating sampling equipment during the course of field investigations.

1.2 Scope/Application

The procedures contained in this document are to be followed when field cleaning sampling equipment, for both re-use in the field, as well as used equipment being returned to the Field Equipment Center (FEC). On the occasion that SESD field investigators determine that any of the procedures described in this section are either inappropriate, inadequate or impractical and that other procedures must be used to clean or decontaminate sampling equipment at a particular site, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on LAN and for maintaining records of review conducted prior to its issuance.

1.4 Definitions

- Decontamination: The process of cleaning dirty sampling equipment to the degree to which it can be re-used, with appropriate QA/QC, in the field.
- Deionized water: Tap water that has been treated by passing through a standard deionizing resin column. At a minimum, the finished water should contain no detectable heavy metals or other inorganic compounds (i.e., at or above analytical detection limits) as defined by a standard inductively coupled Argon Plasma Spectrophotometer (ICP) (or equivalent) scan. Deionized water obtained by other methods is acceptable, as long as it meets the above analytical criteria. Organic-free water may be substituted for deionized water.

- Detergent: A standard brand of phosphate-free laboratory detergent, such as Luminox®.
- Drilling Equipment: All power equipment used to collect surface and sub-surface soil samples or install wells. For purposes of this procedure, direct push is also included in this definition.
- Field Cleaning: The process of cleaning dirty sampling equipment such that it can be returned to the FEC in a condition that will minimize the risk of transfer of contaminants from a site.
- Organic-free water: Tap water that has been treated with activated carbon and deionizing units. At a minimum, the finished water must meet the analytical criteria of deionized water and it should contain no detectable pesticides, herbicides, or extractable organic compounds, and no volatile organic compounds above minimum detectable levels as determined by the Region 4 laboratory for a given set of analyses. Organic-free water obtained by other methods is acceptable, as long as it meets the above analytical criteria.
- Tap water: Water from any potable water supply. Deionized water or organic-free water may be substituted for tap water.

1.5 References

SESD Operating Procedure for Management of Investigation Derived Waste, SESDPROC-202, Most Recent Version

SESD Operating Procedure for Equipment Cleaning and Decontamination at the FEC, SESDPROC-206, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.6 General Precautions

1.6.1 Safety

Proper safety precautions must be observed when field cleaning or decontaminating dirty sampling equipment. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant

requirements, as appropriate. At a minimum, the following precautions should be taken in the field during these cleaning operations:

- When conducting field cleaning or decontamination using laboratory detergent, safety glasses with splash shields or goggles, and latex gloves will be worn.
- No eating, smoking, drinking, chewing, or any hand to mouth contact should be permitted during cleaning operations.

1.6.2 Procedural Precaution

Prior to mobilization to a site, the expected types of contamination should be evaluated to determine if the field cleaning and decontamination activities will generate rinsates and other waste waters that might be considered RCRA hazardous waste or may require special handling.

2 Introduction to Field Equipment Cleaning and Decontamination

2.1 General

The procedures outlined in this document are intended for use by field investigators for cleaning and decontaminating sampling and other equipment in the field. These procedures should be followed in order that equipment is returned to the FEC in a condition that will minimize the risk of transfer of contaminants from a site.

Sampling and field equipment cleaned in accordance with these procedures must meet the minimum requirements for the Data Quality Objectives (DQOs) of the study or investigation. If deviations from these procedures need to be made during the course of the field investigation, they will be documented in the field logbook along with a description of the circumstances requiring the use of the variant procedure.

Cleaning procedures for use at the Field Equipment Center (FEC) are found in SESD Operating Procedure for Equipment Cleaning and Decontamination at the FEC (SESDPROC-206).

2.2 Handling Practices and Containers for Cleaning Solutions

Improperly handled cleaning solutions may easily become contaminated. Storage and application containers must be constructed of the proper materials to ensure their integrity. Following are acceptable materials used for containing the specified cleaning solutions:

- Detergent must be kept in clean plastic, metal, or glass containers until used. It should be poured directly from the container during use.
- Tap water may be kept in tanks, hand pressure sprayers, squeeze bottles, or applied directly from a hose.
- Deionized water must be stored in clean, glass or plastic containers that can be closed prior to use. It can be applied from plastic squeeze bottles.
- Organic-free water must be stored in clean glass or Teflon® containers prior to use. It may be applied using Teflon® squeeze bottles, or with the portable system.

2.3 Disposal of Cleaning Solutions

Procedures for the safe handling and disposition of investigation derived waste (IDW); including used wash water and rinse water are in SESD Operating Procedure for Management of Investigation Derived Waste (SESDPROC-202).

2.4 Sample Collection Equipment Contaminated with Concentrated Materials

Equipment used to collect samples of concentrated materials from investigation sites must be field cleaned before returning from the study. At a minimum, this should consist of washing with detergent and rinsing with tap water. When the above procedure cannot be followed, the following options are acceptable:

1. Leave with facility for proper disposal;
2. If possible, containerize, seal and secure the equipment and leave on-site for later disposal;
3. Containerize, bag or seal the equipment so that no odor is detected and return to the SESD.

It is the project leader's responsibility to evaluate the nature of the sampled material and determine the most appropriate cleaning procedures for the equipment used to sample that material.

2.5 Sample Collection Equipment Contaminated with Environmental Media

Equipment used to collect samples of environmental media from investigation sites should be field cleaned before returning from the study. Based on the condition of the sampling equipment, one or more of the following options must be used for field cleaning:

1. Wipe the equipment clean;
2. Water-rinse the equipment;
3. Wash the equipment in detergent and water followed by a tap water rinse.
4. For grossly contaminated equipment, the procedures set forth in Section 2.4 must be followed.

Under extenuating circumstances such as facility limitations, regulatory limitations, or during residential sampling investigations where field cleaning operations are not feasible, equipment can be containerized, bagged or sealed so that no odor is detected and returned to the FEC without being field cleaned. If possible, FEC personnel should be notified that equipment will be returned without being field cleaned. It is the project leader's responsibility to evaluate the nature of the sampled material and determine the most appropriate cleaning procedures for the equipment used to sample that material.

2.6 Handling of Decontaminated Equipment

After decontamination, equipment should be handled only by personnel wearing clean gloves to prevent re-contamination. In addition, the equipment should be moved away (preferably upwind) from the decontamination area to prevent re-contamination. If the equipment is not to be immediately re-used it should be covered with plastic sheeting or wrapped in aluminum foil to prevent re-contamination. The area where the equipment is kept prior to re-use must be free of contaminants.

3 Field Equipment Decontamination Procedures

3.1 General

Sufficient equipment should be transported to the field so that an entire study can be conducted without the need for decontamination. When equipment must be decontaminated in the field, the following procedures are to be utilized.

3.2 Specifications for Decontamination Pads

Decontamination pads constructed for field cleaning of sampling and drilling equipment should meet the following minimum specifications:

- The pad should be constructed in an area known or believed to be free of surface contamination.
- The pad should not leak.
- If possible, the pad should be constructed on a level, paved surface and should facilitate the removal of wastewater. This may be accomplished by either constructing the pad with one corner lower than the rest, or by creating a sump or pit in one corner or along one side. Any sump or pit should also be lined.
- Sawhorses or racks constructed to hold equipment while being cleaned should be high enough above ground to prevent equipment from being splashed.
- Water should be removed from the decontamination pad frequently.
- A temporary pad should be lined with a water impermeable material with no seams within the pad. This material should be either easily replaced (disposable) or repairable.

At the completion of site activities, the decontamination pad should be deactivated. The pit or sump should be backfilled with the appropriate material designated by the site project leader, but only after all waste/rinse water has been pumped into containers for disposal. See SESD Operating Procedure for Management of Investigation Derived Waste (SESDPROC-202) for proper handling and disposal of these materials. If the decontamination pad has leaked excessively, soil sampling may be required.

3.3 "Classical Parameter" Sampling Equipment

"Classical Parameters" are analyses such as oxygen demand, nutrients, certain inorganic compounds, sulfide, flow measurements, etc. For routine operations involving classical parameter analyses, water quality sampling equipment such as Kemmerers, buckets, dissolved oxygen dunkers, dredges, etc., may be cleaned with the sample water or tap water between sampling locations as appropriate.

Flow measuring equipment such as weirs, staff gages, velocity meters, and other stream gauging equipment may be cleaned with tap water between measuring locations, if necessary.

Note: The procedures described in Section 3.3 are not to be used for cleaning field equipment to be used for the collection of samples undergoing trace organic or inorganic constituent analyses.

3.4 Sampling Equipment used for the Collection of Trace Organic and Inorganic Compounds

For samples undergoing trace organic or inorganic constituent analyses, the following procedures are to be used for all sampling equipment or components of equipment that come in contact with the sample:

1. Clean with tap water and Luminox® detergent using a brush, if necessary, to remove particulate matter and surface films. Equipment may be steam cleaned (Luminox® detergent and high pressure hot water) as an alternative to brushing. Sampling equipment that is steam cleaned should be placed on racks or saw horses at least two feet above the floor of the decontamination pad. PVC or plastic items should not be steam cleaned.
2. Rinse thoroughly with tap water.
3. Rinse thoroughly with organic-free water and place on a clean foil-wrapped surface to air-dry.
4. All equipment must be wrapped with foil. If the equipment is to be stored overnight before it is wrapped in foil, it should be covered and secured with clean, unused plastic sheeting.

3.5 Well Sounders or Tapes

The following procedures are recommended for decontaminating well sounders (water level indicators) and tapes. Unless conditions warrant, it is only necessary to decontaminate the wetted portion of the sounder or tape.

1. Wash with detergent and tap water.
2. Rinse with tap water.
3. Rinse with deionized water.

3.6 Redi-Flo2® Pump

CAUTION – Do not wet the controller. Always disconnect power from the pump when handling the pump body.

The Redi-Flo2® pump and any associated connected hardware (e.g., check valve) should be decontaminated between each monitoring well. The following procedures are required, depending on whether the pump is used solely for purging or used for purging and sampling.

3.6.1 Purge Only (Pump and Wetted Portion of Tubing or Hose)

1. Disconnect power and wash exterior of pump and wetted portion of the power lead and tubing or hose with detergent and water solution.
2. Rinse with tap water.
3. Final rinse with deionized water.
4. Place pump and reel in a clean plastic bag and keep tubing or hose contained in clean plastic or galvanized tub between uses.

3.6.2 Purge And Sample

Grundfos Redi-Flo2® pumps are extensively decontaminated and tested at the FEC to prevent contamination from being transmitted between sites. The relevant sections of SESDPROC-206, *Field Equipment Cleaning and Decontamination at the FEC*, should be implemented in the field where a high risk of cross-contamination exists, such as where NAPL or high-concentration contaminants occur. In most cases, the abbreviated cleaning procedure described below will suffice.

1. Disconnect and discard the previously used sample tubing from the pump. Remove the check valve and tubing adapters and clean separately (See Section 3.6.3 for check valve). Wash the pump exterior with detergent and water.
2. Prepare and fill three containers with decontamination solutions, consisting of Container #1, a tap water/detergent washing solution; Container #2, a tap water rinsing solution; and Container #3, a deionized or organic-free water final rinsing solution. Choice of detergent and final rinsing solution for all steps in this procedure is dependent upon project objectives (analytes and compounds of interest). The containers should be large enough to hold the pump and one to two liters of solution.
3. Place the pump in Container #1. Turn the pump on and circulate the detergent and water solution through the pump and then turn the pump off.
4. Place the pump in Container #2. Turn the pump on and circulate the tap water through the pump and then turn the pump off.
5. Place the pump in Container #3. Turn the pump on and circulate deionized or organic-free water through the pump and then turn the pump off.
6. Disconnect power and remove pump from Container #3. Rinse exterior and interior of pump with fresh deionized or organic-free water.
7. Decontaminate the power lead by washing with detergent and water, followed by tap water and deionized water rinses. This step may be performed before washing the pump if desired.
8. Reassemble check valve and tubing adapters to pump. ALWAYS use Teflon® tape to prevent galling of threads. Firm hand-tightening of fittings or light wrench torque is generally adequate.
9. Place the pump and reel in a clean plastic bag.

3.6.3 Redi-Flo2® Ball Check Valve

1. Remove the ball check valve from the pump head. Check for wear and/or corrosion, and replace as needed. During decontamination check for free-flow in forward direction and blocking of flow in reverse direction.

2. Using a brush, scrub all components with detergent and tap water.
3. Rinse with deionized water.
4. Replace the ball check valve to the Redi-Flo2® pump head.

3.7 Downhole Drilling Equipment

These procedures are to be used for drilling activities involving the collection of soil samples for trace organic and inorganic constituent analyses and for the construction of monitoring wells to be used for the collection of groundwater samples for trace organic and inorganic constituent analyses.

3.7.1 Introduction

Cleaning and decontamination of all equipment should occur at a designated area (decontamination pad) on the site. The decontamination pad should meet the specifications of Section 3.2 of this procedure.

Tap water brought on the site for drilling and cleaning purposes should be contained in a pre-cleaned tank.

A steam cleaner and/or high pressure hot water washer capable of generating a pressure of at least 2500 PSI and producing hot water and/or steam (200° F plus), with a detergent compartment, should be obtained.

3.7.2 Preliminary Cleaning and Inspection

Drilling equipment should be clean of any contaminants that may have been transported from off-site to minimize the potential for cross-contamination. The drilling equipment should not serve as a source of contaminants. Associated drilling and decontamination equipment, well construction materials, and equipment handling procedures should meet these minimum specified criteria:

- All downhole augering, drilling, and sampling equipment should be sandblasted before use if painted, and/or there is a buildup of rust, hard or caked matter, etc., that cannot be removed by steam cleaning (detergent and high pressure hot water), or wire brushing. Sandblasting should be performed prior to arrival on site, or well away from the decontamination pad and areas to be sampled.
- Any portion of the drilling equipment that is over the borehole (kelly bar or mast, backhoe buckets, drilling platform, hoist or chain pulldowns, spindles, cathead, etc.) should be steam cleaned (detergent

and high pressure hot water) and wire brushed (as needed) to remove all rust, soil, and other material which may have come from other sites before being brought on site.

- Printing and/or writing on well casing, tremie tubing, etc., should be removed before use. Emery cloth or sand paper can be used to remove the printing and/or writing. Most well material suppliers can provide materials without the printing and/or writing if specified when ordered. Items that cannot be cleaned are not acceptable and should be discarded.
- Equipment associated with the drilling and sampling activities should be inspected to insure that all oils, greases, hydraulic fluids, etc., have been removed, and all seals and gaskets are intact with no fluid leaks.

3.7.3 Drill Rig Field Cleaning Procedure

Any portion of the drill rig, backhoe, etc., that is over the borehole (kelly bar or mast, backhoe buckets, drilling platform, hoist or chain pulldowns, spindles, cathead, etc.) should be steam cleaned (detergent and high pressure hot water) between boreholes.

3.7.4 Field Decontamination Procedure for Drilling Equipment

The following is the standard procedure for field cleaning augers, drill stems, rods, tools, and associated equipment. This procedure does not apply to well casings, well screens, or split-spoon samplers used to obtain samples for chemical analyses, which should be decontaminated as outlined in Section 3.4 of this procedure.

1. Wash with tap water and detergent, using a brush if necessary, to remove particulate matter and surface films. Steam cleaning (high pressure hot water with detergent) may be necessary to remove matter that is difficult to remove with the brush. Drilling equipment that is steam cleaned should be placed on racks or saw horses at least two feet above the floor of the decontamination pad. Hollow-stem augers, drill rods, etc., that are hollow or have holes that transmit water or drilling fluids, should be cleaned on the inside with vigorous brushing.
2. Rinse thoroughly with tap water.
3. Remove from the decontamination pad and cover with clean, unused plastic. If stored overnight, the plastic should be secured to ensure that it stays in place.

3.7.5 *Field Decontamination Procedure for Direct Push Technology (DPT) Equipment*

1. Certain specific procedures for the decontamination of DPT tools are described in the various sampling procedures, but the following general guidelines apply:
2. Prior to return to the Field Equipment Center, all threaded tool joints should be broken apart and the equipment cleaned per the provisions of *Section 2.5, Sample Collection Equipment Contaminated with Environmental Media* of this procedure.
3. Equipment that contacts the sample media and is cleaned in the field for reuse should be cleaned per the provisions of *Section 3.4, Sampling Equipment used for the Collection of Trace Organic and Inorganic Compounds* of this procedure. This would include piston sampler points and shoes, screen point sampler screens and sheaths, and the drive rods when used for groundwater sampling.
4. Equipment that does not directly contact the sample media and is cleaned in the field for reuse can generally be cleaned per the provisions of *Section 3.7.4, Field Decontamination Procedure for Drilling Equipment* of this procedure.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: Packing, Marking, Labeling and Shipping of Environmental and Waste Samples

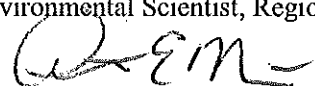
Effective Date: April 20, 2011

Number: SESDPROC-209-R2

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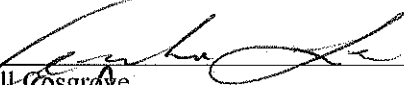
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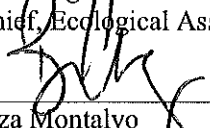
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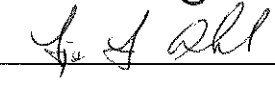
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Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
<p>SESDPROC-209-R2, <i>Packing, Marking, Labeling and Shipping of Environmental and Waste Samples</i>, replaces SESDPROC-209-R1.</p> <p>Title Page: Changed EIB Branch Chief from Antonio Quinones to Archie Lee. Changed Field Quality Manager from Laura Ackerman to Liza Montalvo.</p> <p>Revision History: Previous versions of the document will be maintained by the Document Control Coordinator (DCC). Changed from Field Quality Manager to DCC.</p> <p>Section 1.2: Added the following statement: Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.</p> <p>Section 1.3: Changed requirement so that the DCC is responsible for ensuring the most recent version of the procedure is placed on the SESD LAN and for maintaining records of review conducted prior to its issuance. Deleted reference to the H: drive.</p> <p>Section 3, fifth paragraph: On item #2 on the list, replaced “be sure” with “ensure that.” On item #3, deleted the last sentence that stated that only “Up to three VOA vials may be packed in one Whirl-Pak container or bag.” Deleted outdated item #4 referencing shipment of VOA vials in metal cans. On items #5 and #6, removed references to vermiculite in favor of generic “absorbent material.”</p>	April 20, 2011
<p>SESDPROC-209-R1, <i>Packing, Marking, Labeling and Shipping of Environmental and Waste Samples</i>, replaces SESDPROC-209-R0.</p> <p>General Corrected any typographical, grammatical and/or editorial errors.</p> <p>Title Page Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove’s title from Acting Chief to Chief.</p>	November 1, 2007

<p>Section 1.3 Updated information to reflect that the procedure is located on the H: drive of the LAN. Clarified Field Quality Manager (FQM) responsibilities.</p> <p>Section 1.4 Alphabetized and revised the referencing style for consistency. Added reference to the SHEMP Procedures and Policy Manual.</p> <p>Section 1.5.1 Corrected the title of the Safety, Health, and Environmental Management Program Procedures and Policy Manual.</p>	
<p>SESDPROC-209-R0, Packing, Marking, Labeling and Shipping of Environmental and Waste Samples, Original Issue</p>	<p>February 05, 2007</p>

TABLE OF CONTENTS

1	General Information.....	5
1.1	Purpose.....	5
1.2	Scope/Application	5
1.3	Documentation/Verification.....	6
1.4	References	6
1.5	General Precautions.....	7
1.5.1	<i>Safety</i>	7
2	Shipment of Dangerous Goods	8
3	Shipment of Environmental Samples.....	9

1 General Information

1.1 Purpose

Regulations for packing, marking, labeling, and shipping of dangerous goods by air transport are promulgated by Department of Transportation under 49 CFR, Subchapter C, Hazardous Materials Regulations, and the International Air Transport Authority (IATA), which is equivalent to United Nations International Civil Aviation Organization (UN/ICAO). Transportation of hazardous materials (dangerous goods) by EPA personnel is covered by EPA Order 1000. This document describes general and specific procedures, methods and considerations to be used and observed by SESD field investigators when packing, marking, labeling and shipping environmental and waste samples to ensure that all shipments are in compliance with the above regulations and guidance.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when packing, marking, labeling, and shipping environmental samples and dangerous goods by air transport. Samples collected during field investigations or in response to a hazardous materials incident must be classified prior to shipment, as either environmental or hazardous materials (dangerous goods) samples.

In general, environmental samples include drinking water, most groundwater and ambient surface water, soil, sediment, treated municipal and industrial wastewater effluent, biological specimens, or any samples not expected to be contaminated with high levels of hazardous materials. Samples collected from process wastewater streams, drums, bulk storage tanks, soil, sediment, or water samples from areas suspected of being highly contaminated may require shipment as dangerous goods.

Government employees transporting samples or hazardous materials (i.e., preservatives or waste samples) in government vehicles are not subject to the requirements of this section in accordance with 49 CFR 171.1(d)(5). EPA contractors, however, are not covered by this exemption and may not transport these materials without full compliance with 49 CFR.

Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SEDS management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SEDS local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version.

Title 40 Code of Federal Regulations (CFR), Pt. 136.3, Identification of Test Procedures, July 1, 2001. See Table II, Footnote 3.

Title 49 CFR, Pt. 171.1(d)(5), Applicability of Hazardous Materials Regulations (HMR) to Persons and Functions.

United States Department of Transportation (US DOT). 2003. Letter from Edward T. Mazzullo, Director, Office of Hazardous Materials Standards, to Henry L. Longest II, Acting Assistant Administrator, USEPA, Ref No. 02-0093, February 13, 2003.

US Environmental Protection Agency (US EPA) Order 1000.18, February 16, 1979.

US EPA. 1981. "Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples," Memo from David Weitzman, Work Group Chairman, Office of Occupational Health and Safety (PM-273), April 13, 1981.

US EPA. 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA.

US EPA. Analytical Support Branch Laboratory Operations and Quality Assurance Manual. Region 4 SEDS, Athens, GA, Most Recent Version.

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SEDS, Athens, GA, Most Recent Version.

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when packing, marking, labeling, and shipping environmental or waste samples. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional.

2 Shipment of Dangerous Goods

The project leader is responsible for determining if samples collected during a specific field investigation meet the definitions for dangerous goods. If a sample is collected of a material that is listed in the Dangerous Goods List, Section 4.2, IATA, then that sample must be identified, packaged, marked, labeled, and shipped according to the instructions given for that material. If the composition of the collected sample(s) is unknown, and the project leader knows or suspects that it is a regulated material (dangerous goods), the sample may not be offered for air transport. If the composition and properties of the waste sample or highly contaminated soil, sediment, or water sample are unknown, or only partially known, the sample may not be offered for air transport.

In addition, the shipment of pre-preserved sample containers or bottles of preservatives (e.g., NaOH pellets, HCL, etc.) which are designated as dangerous goods by IATA is regulated. Shipment of nitric acid is strictly regulated. Consult the IATA Dangerous Goods Regulations for guidance. ***Dangerous goods must not be offered for air transport by any personnel except SESD's dangerous goods shipment designee or other personnel trained and certified by IATA in dangerous goods shipment.***

3 Shipment of Environmental Samples

Guidance for the shipment of environmental laboratory samples by personnel is provided in a memorandum dated March 6, 1981, subject "Final National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Laboratory Samples". By this memorandum, the shipment of the following unpreserved samples is not regulated:

- Drinking water
- Treated effluent
- Biological specimens
- Sediment
- Water treatment plant sludge
- POTW sludge

In addition, the shipment of the following preserved samples is not regulated, provided the amount of preservative used does not exceed the amounts found in 40 CFR 136.3 or the USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM), Most Recent Version. This provision is also discussed in correspondence between DOT and EPA (Department of Transportation, Letter from Edward T. Mazzullo, Director, Office of Hazardous Materials Standards, to Henry L. Longest II, Acting Assistant Administrator, USEPA, Ref No.: 02-0093, February 13, 2003). It is the shippers' (individual signing the air waybill) responsibility to ensure that proper amounts of preservative are used:

- Drinking water
- Ambient water
- Treated effluent
- Biological specimens
- Sediment
- Wastewater treatment plant sludge
- Water treatment plant sludge

Samples determined by the project leader to be in these categories are to be shipped using the following protocol, developed jointly between USEPA, OSHA, and DOT. This procedure is documented in the "Final National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Environmental Laboratory Samples."

Untreated wastewater and sludge from Publicly Owned Treatment Works (POTWs) are considered to be "diagnostic specimens" (not environmental laboratory samples). However, because they are not considered to be etiologic agents (infectious) they are not restricted and may be shipped using the procedures outlined below.

Environmental samples should be packed prior to shipment by air using the following procedures:

1. Allow sufficient headspace (ullage) in all bottles (except VOA containers with a septum seal) to compensate for any pressure and temperature changes (approximately 10 percent of the volume of the container).
2. Ensure that the lids on all bottles are tight (will not leak).
3. Place bottles in separate and appropriately sized polyethylene bags and seal the bags. If available, the use of Whirl-Pak bags is preferable, if unavailable seal regular bags with tape (plastic electrical tape).
4. Select a sturdy cooler in good repair. Secure and tape the drain plug with fiber or duct tape inside and outside. Line the cooler with a large heavy duty plastic bag.
5. Place cushioning/absorbent material in the bottom of the cooler and then place the containers in the cooler with sufficient space to allow for the addition of cushioning between the containers.
6. Put "blue ice" (or ice that has been "double bagged" in heavy duty polyethylene bags and properly sealed) on top of and/or between the containers. Fill all remaining space between the containers with absorbent material.
7. Securely fasten the top of the large garbage bag with tape (preferably plastic electrical tape).
8. Place the Chain-of-Custody Record or the CLP Traffic Report Form (if applicable) into a plastic bag, and tape the bag to the inner side of the cooler lid.
9. Close the cooler and securely tape (preferably with fiber tape) the top of the cooler shut. Chain-of-custody seals should be affixed to the top and sides of the cooler within the securing tape so that the cooler cannot be opened without breaking the seal.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: Groundwater Sampling

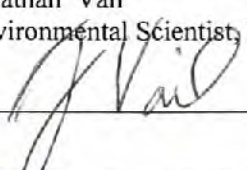
Effective Date: March 6, 2013

Number: SESDPROC-301-R3

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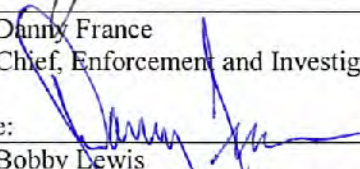
Date:

3/4/2013

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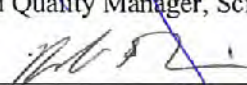
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3/4/13

Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the SESD Document Control Coordinator on the SESD local area network (LAN).

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SESDPROC-301-R0, Groundwater Sampling, Original Issue	February 05, 2007

TABLE OF CONTENTS

1	General Information	6
1.1	Purpose.....	6
1.2	Scope/Application	6
1.3	Documentation/Verification.....	6
1.4	References	6
1.5	General Precautions.....	8
1.5.1	Safety	8
1.5.2	Procedural Precautions.....	8
2	Special Sampling Considerations	10
2.1	Volatile Organic Compounds (VOC) Analysis.....	10
2.2	Special Precautions for Trace Contaminant Groundwater Sampling.....	10
2.3	Sample Handling and Preservation Requirements.....	11
2.4	Quality Control	12
2.5	Records.....	12
3	Groundwater Sampling Methods – Purging	13
3.1	General.....	13
3.2	Purging Methods and Strategies.....	13
3.2.1	Traditional Multiple Volume Purge	13
3.2.1.1	<i>Purging and Purge Adequacy.....</i>	<i>13</i>
3.2.1.1.1	<i>Purge Volume Determination.....</i>	<i>13</i>
3.2.1.1.2	<i>Chemical Parameter Stabilization Criteria</i>	<i>15</i>
3.2.1.1.3	<i>Purge Adequacy Considerations</i>	<i>16</i>
3.2.2	“Tubing-in-Screened-Interval” Method.....	16
3.2.2.1	<i>Purge Criteria.....</i>	<i>17</i>
3.2.2.1.1	<i>Placement of Pump Tubing or Intake</i>	<i>17</i>
3.2.2.1.2	<i>Conditions of Pumping.....</i>	<i>17</i>
3.2.2.1.3	<i>Stability of Chemical Parameters</i>	<i>17</i>

3.3	Equipment Considerations for Purging.....	17
3.3.1	Wells without Plumbing or In-Place Pumps.....	18
3.3.1.1	<i>Purging with Pumps.....</i>	18
3.3.1.1.1	<i>Peristaltic Pumps</i>	18
3.3.1.1.2	<i>Submersible Pumps</i>	19
3.3.1.2	<i>Purging with Bailers</i>	20
3.3.2	Wells with In-Place Plumbing	20
3.3.2.1	<i>Continuously Running Pumps</i>	20
3.3.2.2	<i>Intermittently or Infrequently Running Pumps.....</i>	21
3.3.3	Temporary Monitoring Wells.....	21
3.3.3.1	<i>General Considerations</i>	21
3.3.3.2	<i>Purging When Water Level Is Within Limit of Suction</i>	21
3.3.3.3	<i>Purging When Water Level Is Greater Than Limit of Suction</i>	22
3.3.3.4	<i>Considerations for Direct Push Groundwater Sampling</i>	22
3.4	Field Care of Purging Equipment	22
3.5	Investigation Derived Waste	23
4	Groundwater Sampling Methods – Sampling.....	24
4.1	General.....	24
4.2	Sampling Wells with In-Place Plumbing	24
4.3	Sampling Wells without Plumbing, Within the Limit of Suction	24
4.3.1	Equipment Available	24
4.3.1.1	<i>Peristaltic Pump, Direct from Pump Head Tubing</i>	24
4.3.1.2	<i>Peristaltic Pump/Vacuum Jug.....</i>	25
4.3.1.3	<i>RediFlo2® Electric Submersible Pump (with Teflon® Tubing)</i>	26
4.3.1.4	<i>Bailers.....</i>	26
4.4	Sampling Wells without Plumbing, Exceeding the Limit of Suction	26
4.5	Micro-Purge or No-Purge Sampling Procedures.....	27
4.5.1	Sampling with Pumps	27
4.5.2	HydraSleeves™	27
4.5.3	Passive Diffusion Bags	28
4.5.4	General Considerations for Micro-Purge or No-Purge Sampling	28

4.6	Sample Preservation	28
4.7	Special Sample Collection Procedures	29
4.7.1	Trace Organic Compounds and Metals	29
4.7.2	Order of Sampling with Respect to Analytes	29
4.7.3	Filtering.....	29
4.8	Specific Sampling Equipment Quality Assurance Techniques.....	31
4.9	Auxiliary Data Collection.....	31
4.9.1	Well Pumping Rate – Bucket/Stop Watch Method	31

1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when collecting groundwater samples for field screening or laboratory analysis.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when collecting and handling groundwater samples in the field. On the occasion that SESD field personnel determine that any of the procedures described are either inappropriate, inadequate or impractical and that another procedure must be used to obtain a groundwater sample, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

Columbia Analytical Services, Lab Science News, Passive Diffusion Devices & Polyethylene Diffusion Bag (PDB) Samplers.

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version

Puls, Robert W., and Michael J. Barcelona. 1989. Filtration of Ground Water Samples for Metals Analysis. Hazardous Waste and Hazardous Materials 6(4), pp.385-393.

Puls, Robert W., Don A. Clark, and Bert Bledsoe. 1992. Metals in Ground Water: Sampling Artifacts and Reproducibility. Hazardous Waste and Hazardous Materials 9(2), pp. 149-162.

SESD Guidance Document, Design and Installation of Monitoring Wells, SESDGUID-001, Most Recent Version

SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version

SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

SESD Operating Procedure for Field pH Measurement, SESDPROC-100, Most Recent Version

SESD Operating Procedure for Field Specific Conductance Measurement, SESDPROC-101, Most Recent Version

SESD Operating Procedure for Field Temperature Measurement, SESDPROC-102, Most Recent Version

SESD Operating Procedure for Field Turbidity Measurement, SESDPROC-103, Most Recent Version

SESD Operating Procedure for Groundwater Level and Well Depth Measurement, SESDPROC-105, Most Recent Version

SESD Operating Procedure for Management of Investigation Derived Waste, SESDPROC-202, Most Recent Version

SESD Operating Procedure for Pump Operation, SESDPROC-203, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, SESDPROC-206, Most Recent Version

SESD Operating Procedure for Potable Water Supply Sampling, SESDPROC-305, Most Recent Version

The Interstate Technology & Regulatory Council, Technology Overview of Passive Sampler Technologies, Prepared by The Interstate Technology & Regulatory Council Diffusion Sampler Team, March 2006.

United States Environmental Protection Agency (US EPA). 1975. Handbook for Evaluating Water Bacteriological Laboratories. Office of Research and Development (ORD), Municipal Environmental Research Laboratory, Cincinnati, Ohio.

US EPA. 1977. Sampling for Organic Chemicals and Microorganisms in the Subsurface. EPA-600/2-77/176.

US EPA. 1978. Microbiological Methods for Monitoring the Environment, Water and Wastes. ORD, Municipal Environmental Research Laboratory, Cincinnati, Ohio.

US EPA. 1981. "Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples," Memo from David Weitzman, Work Group Chairman, Office of Occupational Health and Safety (PM-273), April 13, 1981.

US EPA. 1995. Ground Water Sampling - A Workshop Summary. Proceedings from the Dallas, Texas November 30 – December 2, 1993 Workshop. ORD, Robert S. Kerr Environmental Research Laboratory. EPA/600/R-94/205, January 1995.

US EPA. Analytical Support Branch Laboratory Operations and Quality Assurance Manual. Region 4 SEDS, Athens, GA, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SEDS, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when collecting groundwater samples. Refer to the SEDS Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines should be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when collecting groundwater samples.

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Always sample from the anticipated cleanest, i.e., least contaminated location, to the most contaminated location. This minimizes the opportunity for cross-contamination to occur during sampling.
- Collected samples must remain in the custody of the sampler or sample custodian until the samples are relinquished to another party.

- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.
- Documentation of field sampling is done in a bound logbook.
- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- All shipping documents, such as air bills, bills of lading, etc., shall be retained by the project leader and placed in the project files.

2 Special Sampling Considerations

2.1 Volatile Organic Compounds (VOC) Analysis

Groundwater samples for VOC analysis must be collected in 40 ml glass vials with Teflon® septa. The vial may be either preserved with concentrated hydrochloric acid or they may be unpreserved. Preserved samples have a two-week holding time, whereas unpreserved samples have only a seven-day holding time. In the great majority of cases, the preserved vials are used to take advantage of the extended holding time. In some situations, however, it may be necessary to use the unpreserved vials. For example, if the groundwater has a high amount of dissolved limestone, i.e., is highly calcareous, there will most likely be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles. This will render the sample unacceptable. In this case, unpreserved vials should be used and arrangements must be confirmed with the laboratory to ensure that they can accept the unpreserved vials and meet the shorter sample holding times.

The samples should be collected with as little agitation or disturbance as possible. The vial should be filled so that there is a meniscus at the top of the vial and absolutely no bubbles or headspace should be present in the vial after it is capped. After the cap is securely tightened, the vial should be inverted and tapped on the palm of one hand to see if any undetected bubbles are dislodged. If a bubble or bubbles are present, the vial should be topped off using a minimal amount of sample to re-establish the meniscus. Care should be taken not to flush any preservative out of the vial during topping off. If, after topping off and capping the vial, bubbles are still present, a new vial should be obtained and the sample re-collected.

Samples for VOC analysis must be collected using either stainless steel or Teflon® equipment, such as:

- Bailers must be constructed of stainless steel or Teflon®
- RediFlo2® submersible pumps used for sampling should be equipped with Teflon® sample delivery tubing
- Peristaltic pump/vacuum jug assemblies should be outfitted with Teflon® tubing from the water column to the transfer cap, which should also be constructed of Teflon®

2.2 Special Precautions for Trace Contaminant Groundwater Sampling

- A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- Sample containers for samples suspected of containing high concentrations of contaminants shall be stored separately.

- Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area if sampling devices are to be reused. Samples of waste or highly contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.
- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other members collect the samples.
- Clean plastic sheeting will be placed on the ground at each sample location to prevent or minimize contaminating sampling equipment by accidental contact with the ground surface.
- Samplers must use new, verified certified-clean disposable or non-disposable equipment cleaned according to procedures contained in SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206) for collection of samples for trace metals or organic compound analyses.

2.3 Sample Handling and Preservation Requirements

1. Groundwater samples will typically be collected from the discharge line of a pump or from a bailer, either from the pour stream of an up-turned bailer or from the stream from a bottom-emptying device. Efforts should be made to reduce the flow from either the pump discharge line or the bailer during sample collection to minimize sample agitation.
2. During sample collection, make sure that the pump discharge line or the bailer does not contact the sample container.
3. Place the sample into appropriate, labeled containers. Samples collected for VOC, acidity and alkalinity analysis must not have any headspace. All other sample containers must be filled with an allowance for ullage.
4. All samples requiring preservation must be preserved as soon as practically possible, ideally immediately at the time of sample collection. If preserved VOC vials are used, these will be preserved with concentrated hydrochloric acid by ASB personnel prior to departure for the field investigation. For all other chemical preservatives, SESD will use the appropriate chemical preservative generally stored in an individual single-use vial as described in the SESD Operating Procedure for Field Sampling Quality Control (SESDPROC-011). The adequacy of sample preservation will be checked after the addition of the preservative for all samples except for the samples collected for VOC analysis. If additional preservative is needed, it should be added to achieve adequate preservation. Preservation requirements for groundwater samples are found in the USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM).

2.4 Quality Control

If possible, a control sample should be collected from a location not affected by the possible contaminants of concern and submitted with the other samples. This control sample should be collected as close to the sampled area as possible and from the same water-bearing formation. Equipment blanks should be collected if equipment is field cleaned and re-used on-site or if necessary to document that low-level contaminants were not introduced by pumps, bailers or other sampling equipment.

2.5 Records

Information generated or obtained by SESD personnel will be organized and accounted for in accordance with SESD records management procedures found in SESD Operating Procedure for Control of Records, SESDPROC-002. Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation in accordance with SESD Operating Procedure for Logbooks, SESDPROC-010 and SESD Procedure for Sample and Evidence Management, SESDPROC-005.

3 Groundwater Sampling Methods – Purging

3.1 General

Purging is the process of removing stagnant water from a well, immediately prior to sampling, causing its replacement by groundwater from the adjacent formation that is representative of actual aquifer conditions. In order to determine when a well has been adequately purged, field investigators should monitor, at a minimum, the pH, specific conductance and turbidity of the groundwater removed during purging and, in the case of permanent monitoring wells, observe and record the volume of water removed.

There are several purging strategies that may be used, depending on specific conditions encountered for given well sampling situations. When a specific well is characterized, based on the field investigators experience and knowledge, as having fairly typical water levels, depths and purge volumes, as determined according to the procedures in Section 3.2.1, below, SESD will normally use the multiple volume purging procedures and equipment described in Sections 3.2.1 and 3.3 of this procedure for purging the well.

When the traditional multiple volume purge method is considered and it is determined that excessive quantities of IDW would be generated using this method, it may be appropriate, under very limited and specific circumstances, to use an alternate method that reduces the time and amount of purge water to be removed prior to sampling the well. The field project leader will select the alternate method only after careful consideration of the conditions presented by the well and the impact these conditions have on all aspects of the sampling event (time required to sample, quantities of IDW requiring management, etc.).

The alternate purge procedures or sampling strategies available are the “Tubing-in-Screened Interval” method and the MicroPurge or No-Purge methods. These are described and discussed in Sections 3.2.2 and 4.5 of this operating procedure, respectively.

3.2 Purging Methods and Strategies

3.2.1 Traditional Multiple Volume Purge

3.2.1.1 Purging and Purge Adequacy

3.2.1.1.1 Purge Volume Determination

Prior to initiating the purge, the amount of water standing in the water column (water inside the well riser and screen) should be determined, if possible. To do this, the diameter of the well should be determined and the water level and total depth of the well should be measured and recorded. Specific methodology for obtaining these measurements is found in SESD Operating Procedure for Groundwater Level and Well Depth Measurement (SESDPROC-105).

Once this information is obtained, the volume of water to be purged can be determined using one of several methods. One is the equation:

$$V = 0.041 d^2 h$$

Where: h = depth of water in feet

d = diameter of well in inches

V = volume of water in gallons

Alternatively, the volume of standing water in the well and the volume of three water columns may be determined using a casing volume per foot factor for the appropriate diameter well, similar to that in Table 3.2.1. The water level is subtracted from the total depth, providing the length of the water column. This length is multiplied by the appropriate factor in the Table 3.2.1, corresponding to either the single well volume or the triple well volume, to determine both the single well volume and triple well volumes, in gallons, for the well in question. Other acceptable methods include the use of nomographs or other equations or formulae.

TABLE 3.2.1: WELL CASING DIAMETER VOLUME FACTORS

Casing Diameter (inches)	Gallons/ft, One Water Column	Gallons/ft, Three Water Columns
1	0.04	0.12
2	0.16	0.48
3	0.37	1.11
4	0.65	1.98
5	1.02	3.06
6	1.47	4.41
7	1.99	5.97
8	2.61	7.83
9	3.30	9.90
10	4.08	12.24
11	4.93	14.79
12	5.87	17.61

With respect to volume, an adequate purge is normally achieved when three to five well volumes have been removed. The field notes should reflect the single well volume calculations or determinations, according to one of the above methods, and a reference to the appropriate

multiplication of that volume, i.e., a minimum three well volumes, clearly identified as a purge volume goal.

3.2.1.1.2 Chemical Parameter Stabilization Criteria

With respect to the ground water chemistry, an adequate purge is achieved when the pH and specific conductance of the ground water have stabilized and the turbidity has either stabilized or is below 10 Nephelometric Turbidity Units (NTUs) (twice the Primary Drinking Water Standard of 5 NTUs). Although 10 NTUs is normally considered the minimum goal for most ground water sampling objectives, lower turbidity has been shown to be easily achievable in most situations and reasonable attempts should be made to achieve these lower levels. (Note: Because groundwater temperature is subject to rapid changes when collected for parameter measurement, its usefulness is subject to question for the purpose of determining parameter stability. As such, it has been removed from the list of parameters used for stability determination. Even though temperature is not used to determine stability during well purging, it is still advisable to record the sample temperature, along with the other groundwater chemistry parameters during well purging, as it may be needed to interpret other chemical parameter results in some situations.)

Stabilization occurs when, for at least three consecutive measurements, the pH remains constant within 0.1 Standard Unit (SU) and specific conductance varies no more than approximately 5 percent. Other parameters, such as dissolved oxygen (DO), may also be used as a purge adequacy parameter. Normal goals for DO are 0.2 mg/L or 10% saturation, whichever is greater. DO measurements must be conducted using either a flow-through cell or an over-topping cell to minimize or reduce any oxygenation of the sample during measurement. Oxidation Reduction Potential (ORP) should not be used as a purge stabilization parameter but may be measured during purging to obtain the measurement of record for ORP for the sampling event.

There are no set criteria for establishing how many total sets of measurements are adequate to document stability of parameters. If the calculated purge volume is small, the measurements should be taken frequently enough to provide a sufficient number of measurements to evaluate stability. If the purge volume is large, measurements taken every 15 minutes, for example, may be sufficient. See the SESD Operating Procedures for Field pH Measurement (SESDPROC-100), Field Specific Conductance Measurement (SESDPROC-101), Field Temperature Measurement (SESDPROC-102), Field Turbidity Measurement (SESDPROC-103), Field Measurement of Dissolved Oxygen (SESDPROC-106) and Field Measurement of Oxidation-Reduction Potential (SESDPROC-113) for procedures for conducting these measurements.

If, after three well volumes have been removed, the chemical parameters have not stabilized according to the above criteria, additional well volumes (up to five well volumes), should be removed. If the parameters have not stabilized within five volumes, it is at the discretion of the project leader whether or not to collect a sample or to continue purging. If, after five well volumes, pH and conductivity have stabilized and the turbidity is still decreasing and approaching an acceptable level, additional purging should be considered to obtain the best sample possible, with respect to turbidity. The conditions of sampling should be noted in the field log.

3.2.1.1.3 Purge Adequacy Considerations

In some situations, even with slow purge rates, a well may be pumped or bailed dry (evacuated). In these situations, this generally constitutes an adequate purge and the well can be sampled following sufficient recovery (enough volume to allow filling of all sample containers). ***It is not necessary that the well be evacuated three times before it is sampled.*** The pH, specific conductance, temperature, and turbidity should be measured and recorded, during collection of the sample from the recovered volume, as the measurements of record for the sampling event.

For wells with slow recovery, attempts should be made to avoid purging them to dryness. This can be accomplished, for example, by slowing the purge rate. As water enters a well that has been purged to dryness, it may cascade down the sand pack and/or the well screen, stripping volatile organic constituents that may be present and/or introducing soil fines into the water column.

It is particularly important that wells be sampled as soon as possible after purging. If adequate volume is available immediately upon completion of purging, the well must be sampled immediately. If not, sampling should occur as soon as adequate volume has recovered. If possible, sampling of wells which have a slow recovery should be scheduled so that they can be purged and sampled in the same day, after adequate volume has recovered. Wells of this type should, unless it is unavoidable, not be purged at the end of one day and sampled the following day.

3.2.2 “Tubing-in-Screened-Interval” Method

The “Tubing-in-Screen” method, sometimes referred to as the “Low Flow” method, is used primarily when calculated purge volumes for the traditional purging method are excessive and present issues related to timely completion of the project and/or management of investigation derived waste.

3.2.2.1 Purge Criteria

3.2.2.1.1 Placement of Pump Tubing or Intake

The peristaltic pump tubing or intake point of the submersible pump is placed in the approximate mid-portion of the screened interval of the well. By definition, this method cannot be applied for purging with a bailer.

3.2.2.1.2 Conditions of Pumping

Prior to initiation of pumping, a properly decontaminated well sounder should be lowered into the well being sampled to monitor the static water level prior to and during the purging process. Ideally, there should be only a slight and stable drawdown of the water column after pumping begins. If this condition cannot be met, then one of the other methods should be employed.

3.2.2.1.3 Stability of Chemical Parameters

As with the traditional purging method described in Section 3.2.1, it is important that all chemical parameters be stable as defined in Section 3.2.1.1 prior to sampling.

3.3 Equipment Considerations for Purging

Monitoring well purging is accomplished by using in-place plumbing and dedicated pumps or by using portable pumps/equipment when dedicated systems are not present. The equipment utilized by Branch personnel will usually consist of peristaltic pumps and variable speed electric submersible pumps, but may also include bladder pumps or inertial pumps. The pump of choice is usually a function of the well diameter, the depth to water, the depth of the well and the amount of water that is to be removed during purging. Whenever the head difference between the sampling location and the water level is less than the limit of suction and the volume to be removed is reasonably small, a peristaltic pump should be used for purging. For wells where the water level is below the limit of suction (approximately 25' to 30', and/or where there is a large volume of water to be purged), the variable speed electric submersible pump would be the pump of choice. SESD Operating Procedure for Pump Operation (SESDPROC-203) contains the use and operating instructions for all pumps commonly used during SESD ground water investigations.

Bailers may also be used for purging in appropriate situations, however, their use is discouraged. Bailers tend to disturb any sediment that may be present in the well, creating or increasing sample turbidity. Bailers, if improperly used, may also strip volatile organic compounds from the water column being sampled. If a bailer is used, it should be a closed-top Teflon® bailer.

3.3.1 Wells Without Plumbing or In-Place Pumps

For permanent monitoring wells, the depth to water (water level) and depth of the well (total depth) should be determined before purging. Caution should be exercised during this procedure to prevent cross-contamination between wells. This is a critical concern when samples for trace organic compounds or metals analyses are collected. See SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) for cleaning procedures for well sounders. After cleaning, the well sounding device should be protected to keep it clean until its next use.

3.3.1.1 Purging with Pumps

3.3.1.1.1 Peristaltic Pumps

The following step-by-step procedures describe the process of purging with a peristaltic pump:

1. Cut a length of standard-cleaned (SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206)) Teflon® tubing, equal to the well depth plus an additional five to ten feet. Enough tubing is needed to run from the ground surface up to the top of the well casing and back down to the bottom of the well. This will allow for operation of the pump at all possible water level conditions in the well.
2. Place one end of the tubing into the vacuum side of the peristaltic pump head. Proper sizing of the Teflon® and Silastic® or Tygon® tubing should allow for a snug fit of the Teflon® tubing inside the flexible tubing mounted in the pump head.
3. Run a short section of tubing (does not have to be Teflon®) from the discharge side of the pump head to a graduated bucket.
4. Place the free end of the Teflon® tubing into the well until the end of the tubing is just below the surface of the water column.
5. Secure the Teflon® tubing to the well casing or other secure object using electrician's tape or other suitable means. This will prevent the tubing from being lost in the well should the tubing detach from the pump head.
6. Turn on the pump to produce a vacuum on the well side of the pump head and begin the purge. Observe pump direction to ensure that a vacuum is being applied to the purge line. If the purge line is being pressurized, either switch the tubing at the pump head or reverse the polarity of the cables on the pump or on the battery.

7. If the pumping rate exceeds the recovery rate of the well, continue to lower the tubing into the well, as needed, until the drawdown stabilizes or the well is evacuated to dryness. If the pump is a variable speed peristaltic pump, and the water level in the well is being drawn down, reduce the speed of the pump in an attempt to stabilize the drawdown. If the well can be purged without evacuating the well to dryness, a sample with greater integrity can be obtained.
8. For wells which are not evacuated to dryness, particularly those with recovery rates equal to or very nearly equal to the purge rate, there may not be a complete exchange and removal of stagnant water in that portion of the water column above the tubing intake. For this reason, it is important that the tubing intake be placed in the very uppermost portion of the water column while purging. Standard field measurements should frequently be taken during this process to verify adequacy of the purge and readiness for sampling, as described in Section 3.

3.3.1.1.2 Submersible Pumps

When a submersible pump is used for well purging, the pump itself is lowered into the water column. The pump must be cleaned as specified in SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205).

The pump/hose assembly used in purging should be lowered into the top of the standing water column and not deep into the column. This is done so that the purging will "pull" water from the formation into the screened area of the well and up through the casing so that the entire static volume can be removed. If the pump is placed deep into the water column, the water above the pump may not be removed, and the subsequent samples, particularly if collected with a bailer, may not be representative of the aquifer conditions. It is recommended that the pump not be lowered more than three to five feet into the water column. If the recovery rate of the well is faster than the pump rate and no observable draw down occurs, the pump should be raised until the intake is within one foot of the top of the water column for the duration of purging. If the pump rate exceeds the recovery rate of the well, the pump will have to be lowered, as needed, to accommodate the drawdown. After the pump is removed from the well, the hose and the pump should be cleaned as outlined in SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205).

3.3.1.2 Purging with Bailers

Standard-cleaned (SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206)), closed top Teflon® bailers with Teflon® coated stainless steel leaders and new nylon rope are lowered into the top of the water column, allowed to fill, and removed. It is critical that bailers be slowly and gently immersed into the top of the water column, particularly during final stages of purging, to minimize turbidity and disturbance of volatile organic constituents. The use of bailers for purging and sampling is discouraged because the correct technique is highly operator dependent and improper use may result in an unrepresentative sample.

3.3.2 Wells With In-Place Plumbing

Wells with in-place plumbing are commonly found at municipal water treatment plants, industrial water supplies, private residences, etc. Many permanent monitoring wells at active facilities are also equipped with dedicated, in-place pumps. The objective of purging wells with in-place pumps is the same as with monitoring wells without in-place pumps, i.e., to ultimately collect a ground water sample representative of aquifer conditions. Among the types of wells identified in this section, two different approaches are necessary.

A permanent monitoring well with an in-place pump should, in all respects, be treated like a monitoring well without a pump. One limitation is that in most cases the in-place pump is “hard” mounted, that is, the pump is suspended in the well at a pre-selected depth and cannot be moved up or down during purging and sampling. In these cases, well volumes are calculated, parameters are measured and the well is sampled from the pump discharge, after volume removal and parameter conditions have been met.

In the case of the other types of wells, i.e., municipal, industrial and residential supply wells, however, not enough is generally known about the construction aspects of the wells to apply the same criteria as used for monitoring wells, i.e., 3 to 5 well volumes. The volume to be purged in these situations, therefore, depends on several factors: whether the pumps are running continuously or intermittently and whether or not any storage/pressure tanks are located between the sampling point and the pump. The following considerations and procedures should be followed when purging wells with in-place plumbing under the conditions described.

3.3.2.1 Continuously Running Pumps

If the pump runs more or less continuously, no purge (other than opening a valve and allowing it to flush for a few minutes) is necessary. If a storage tank is present, a spigot, valve or other sampling point should be located between the

pump and the storage tank. If not, locate the valve closest to the tank. Measurements of pH, specific conductance, temperature, and turbidity are recorded at the time of sampling.

3.3.2.2 Intermittently or Infrequently Running Pumps

If the pump runs intermittently or infrequently, best judgment should be utilized to remove enough water from the plumbing to flush standing water from the piping and any storage tanks that might be present. Generally, under these conditions, 15 to 30 minutes will be adequate. Measurements of pH, specific conductance, temperature and turbidity should be made and recorded at intervals during the purge and the final measurements made at the time of sampling should be considered the measurements of record for the event.

3.3.3 Temporary Monitoring Wells

3.3.3.1 General Considerations

Procedures used to purge temporary ground water monitoring wells differ from permanent wells because temporary wells are installed for immediate sample acquisition. Wells of this type may include standard well screen and riser placed in boreholes created by hand augering, power augering, or by drilling. They may also consist of a rigid rod and screen that is pushed, driven, or hammered into place to the desired sampling interval, such as a direct push Wellpoint®, a Geoprobe® Screen Point 15/16 sampler or a Hydropunch® sampler. As such, the efforts to remove several volumes of water to replace stagnant water do not necessarily apply because stagnant water is not present. It is important to note, however, that the longer a temporary well is in place and not sampled, the more stagnant the water column becomes and the more appropriate it becomes to apply, to the extent possible, standard permanent monitoring well purging criteria to it to re-achieve aquifer conditions.

In cases where the temporary well is to be sampled immediately after installation, purging is conducted primarily to mitigate the impacts of installation. In most cases, temporary well installation procedures disturb the existing aquifer conditions, resulting primarily in increased turbidity. Therefore, the goal of purging is to reduce the turbidity and remove the volume of water in the area directly impacted by the installation procedure. Low turbidity conditions in these types of wells that are completed within the limit of suction are typically and routinely achieved by the use of low-flow/low stress purging techniques using variable speed peristaltic pumps.

3.3.3.2 Purging When Water Level Is Within Limit of Suction

In situations where the elevation of the top of the water column is within the limit of suction (no greater than about 25 feet head difference between the pump and the water level), a variable speed peristaltic pump may be used to purge

temporary wells. Enough tubing is deployed to reach the bottom of the temporary well screen. At the onset of purging, the tubing is slowly lowered to the bottom of the screen and is used to remove any formation material which may have entered the well screen during installation. This is critical to ensuring rapid achievement of low turbidity conditions. After the formation material is removed from the bottom of the screen, the tubing is slowly raised through the water column to near the top of the column. The tubing can be held at this level to determine if the pump rate is drawing down the water level in the well. If the water level remains the same, secure the tubing at the surface to maintain this pumping level.

If drawdown is observed on initiation of pumping, reduce the pump speed and attempt to match the drawdown of the well. Sustained pumping at these slow rates will usually result in a relatively clear, low turbidity sample. If the drawdown stabilizes, maintain that level, however, if it continues to lower, "chase" the water column until the well is evacuated. In this case, the recovered water column may be relatively free of turbidity and can be sampled. It may take several episodes of recovery to provide enough volume for a complete sample.

3.3.3.3 Purging When Water Level Is Greater Than Limit of Suction

In situations where the elevation of the water table is greater than the limit of suction, peristaltic pumps cannot be used to purge temporary wells. If the temporary well is a ScreenPoint15® sampler with small diameter probe rod riser, the only practical choices for water removal are a small diameter bailer, a small diameter bladder pump or an inertial pump. If the well is to be used strictly for VOC screening, it may be acceptable to use the bailer to bail as much sediment from the well as possible prior to sampling. If metals are the analytes of concern, the bladder pump is the best choice for lowering the turbidity of the water column prior to sampling, followed next by the inertial pump. For larger diameter temporary wells, two-inch diameter or greater, bailers and the Grundfos® RediFlo2 may be used although excessive silt or other "fines" may present problems with the operation of the pump.

3.3.3.4 Considerations for Direct Push Groundwater Sampling

With many of the direct push sampling techniques, purging is either not practical or possible, therefore, no purging is conducted. The sampling device is simply pushed or driven to the desired depth and opened and the sample is collected and retrieved. As a result, some samples collected in this way may not be satisfactory or acceptable for certain analyses, i.e., the subject procedure may yield a turbid sample that is not appropriate for metals analyses.

3.4 Field Care of Purging Equipment

New plastic sheeting should be placed on the ground surface around the well casing to prevent contamination of the pumps, hoses, ropes, etc., in the event they accidentally

come into contact with the ground surface or, for some reason, they need to be placed on the ground during the purging event. It is preferable that hoses used in purging that come into contact with the ground water be kept on a spool or contained in a large wash tub lined with plastic sheeting, both during transportation and during field use, to further minimize contamination by the transporting vehicle or the ground surface.

Careful consideration shall be given to using submersible pumps to purge wells which are excessively contaminated with oily compounds, because it may be difficult to adequately decontaminate severely contaminated pumps under field conditions. When wells of this type are encountered, alternative purging methods, such as bailers, should be considered.

3.5 Investigation Derived Waste

Purging generates quantities of purge water or investigation derived waste (IDW), the disposition of which must be considered. See SESD Operating Procedure for Management of Investigation Derived Waste (SESDPROC-202) for guidance on management or disposal of this waste.

4 Groundwater Sampling Methods – Sampling

4.1 General

Sampling is the process of obtaining, containerizing, and preserving (if required) a ground water sample after the purging process is complete. Non-dedicated pumps for sample collection generally should not be used. Many pumps are made of materials such as brass, plastic, rubber, or other elastomeric products which may cause chemical interferences with the sample. Their principle of operation may also render them unacceptable as a sample collection device. It is recognized that there are situations, such as industrial or municipal supply wells or private residential wells, where a well may be equipped with a dedicated pump from which a sample would not normally be collected. Discretion should always be used in obtaining a sample.

4.2 Sampling Wells With In-Place Plumbing

Samples should be collected following purging from a valve or cold water tap as near to the well as possible, preferably prior to any storage/pressure tanks or physical/chemical treatment system that might be present. Remove any hose that may be present before sample collection and reduce the flow to a low level to minimize sample disturbance, particularly with respect to volatile organic constituents. Samples should be collected directly into the appropriate containers as specified in the ASBLOQAM. It may be necessary to use a secondary container, such as a clean 8 oz. or similar size sample jar or a stainless steel scoop, to obtain and transfer samples from spigots with low ground clearance. Also, refer to the discussion in the SESD Operating Procedure for Potable Water Supply Sampling (SESDPROC-305), Sec. 4.2, Potable Water Samples Collected from Wells with In-Place Plumbing. Potable well measurements for pH, specific conductance and turbidity and possibly temperature, if warranted, should be recorded at the time of sample collection.

4.3 Sampling Wells Without Plumbing, Within the Limit of Suction

4.3.1 Equipment Available

The pump of choice for sampling ground water within the limit of suction is the variable-speed peristaltic pump. Its use is described in the following sections. Other acceptable alternatives that may be used under these conditions are the RediFlo2® electric submersible pump (with Teflon® tubing) and a closed-top Teflon® bailer.

4.3.1.1 Peristaltic Pump, Direct from Pump Head Tubing

Samples for some constituents, primarily inorganic analytes such as metals and cyanide, may be collected directly from the pump head tubing. This method is acceptable under the following conditions:

- The pump head tubing must be changed between sampling locations;

- The pump head tubing must be either be certified clean according to SESD's internal quality control program described in Section 3.2 of the SESD Operating Procedure for Field Sampling Quality Control (SESDPROC-011) or
- An equipment rinsate blank is collected by pumping de-ionized water through a piece of the tubing.

4.3.1.2 Peristaltic Pump/Vacuum jug

It is not acceptable to collect samples for organic compound analyses through the flexible tubing used in the pump head. When collecting samples for organic compound analyses it is necessary to use a vacuum container, placed between the pump and the well for sample collection. The following step-by-step procedures describe the process of sampling with a peristaltic pump and vacuum jug (see note following these procedures for collection of VOC samples):

1. Disconnect the purge tubing from the pump. Make sure the tubing is securely attached to the protective casing or other secure object.
2. Insert the tubing into one of the ferrule nut fittings of a Teflon® vacuum container transfer cap assembly.
3. Place a suitable length of Teflon® tubing between the remaining transfer cap assembly ferrule nut fitting and the vacuum side of the flexible tubing in the peristaltic pump head. Securely hand-tighten both fittings.
4. Turn the pump on. Water should begin to collect in the transfer container (typically a 1-liter sample container) within a few minutes. If water does not begin to flow into the container within several minutes, check the transfer cap fittings and make sure the assembly is tightly attached to the container. It may be necessary to tighten the ferrule nuts with a wrench or pliers to achieve a vacuum in the system, particularly when approaching the maximum head difference between the pump and water table (limit of suction).
5. When the transfer container is nearly full, turn off the pump, remove the transfer cap assembly, and pour the sample into the appropriate containers. Because the 1-liter containers used by the Branch are rinsed with nitric acid during cleaning, they cannot be used for collecting samples to be analyzed for nitrogen sensitive parameters.
6. If additional sample volume is needed, replace the transfer cap assembly, turn the pump on, and collect additional volume. The use of Teflon® valves or ball check devices to retain the water column in the sample delivery tubing during the transfer phase, when large volumes of sample are required, is acceptable. These devices, however, must be constructed so that they may be completely disassembled and cleaned according to the procedures in SESD

7. When sampling is completed, all Teflon® tubing should be discarded.

NOTE: Samples for volatile organic compound analyses cannot be collected using this method. If samples for VOC analyses are required, they must be collected with a Teflon® or stainless steel bailer or by other approved methods, such as the “soda straw” method. The “soda straw” method involves allowing the tubing to fill, by either lowering it into the water column (A) or by filling it via suction applied by the pump head (B). If method (A) is used, the tubing is removed from the well after filling and the captured sample is allowed to drain into the sample vial. If method (B) is used, after running the pump and filling the tubing with sample, the pump speed is reduced and the direction reversed to push the sample out of the tubing into the vials. Avoid completely emptying the tubing when filling the sample vials when using method (B) to prevent introducing water that was in contact with the flexible pump head tubing. Either method is repeated, as necessary, until all vials are filled.

4.3.1.3 RediFlo2® Electric Submersible Pump (with Teflon® Tubing)

After purging has been accomplished with RediFlo2® electric submersible pump, the sample may be obtained directly from the pump discharge, provided that Teflon® tubing was used for the sample delivery line. The discharge rate of the pump should be reduced during volatile organic compound sample collection to minimize sample disturbance. Note, if the RediFlo2® electric submersible pump is used for sampling, the pump must undergo a full external and internal cleaning. In addition, pump rinsate blanks must be collected, at the appropriate frequency, to demonstrate that the pump has been adequately cleaned between wells.

4.3.1.4 Bailers

New bailer rope should be attached to the bailer via a Teflon® coated stainless steel wire. (If a bailer was used to purge the well, it may also be used to sample the well and new bailer rope is not required between purging and sampling). The bailer should be gently immersed in the top of the water column until just filled. At this point, the bailer should be slowly removed and the contents emptied into the appropriate sample containers.

4.4 Sampling Wells without Plumbing, Exceeding the Limit of Suction

All methods described previously in Section 4.3.2.1.3, RediFlo2® Electric Submersible Pumps, and Section 4.3.2.1.4, Bailers, are suitable sample methods where the water table is too deep to consider the use of a peristaltic pump for sampling.

4.5 Micro-Purge or No Purge Sampling Procedures

The Micro-Purge or No Purge sampling procedures are usually employed when it necessary to keep purge volumes to an absolute minimum. Among the Micro-Purge or No Purge procedures that might be employed are:

- Low pump rate sampling with peristaltic or submersible pumps (typical Micro-Purge sampling),
- HydraSleeve™ or
- Passive diffusion bag (PDB) sampling

The use of these procedures is acceptable only when the site hydrogeology is well understood, with respect to the hydraulic conductivity of geologic materials within the well screen interval. The underlying assumption, when employing these procedures, is that the formation in which the well is screened has a high hydraulic conductivity ($K > 10^{-5}$ cm/sec, for example), resulting in a state of equilibrium existing between the water standing in the screened interval and the formation water in which the well is screened. In this situation, the well is considered to be in a perpetually “purged” state and purging is not required.

These procedures are generally impractical for SESD to implement because of the general lack of hydrogeologic information for the sampled wells and the real necessity, in some cases, that the pumps be pre-deployed to overcome issues related to turbidity resulting from pump placement prior to sampling.

4.5.1 Sampling with Pumps

The peristaltic pump tubing or intake point of the submersible pump is placed in the approximate mid-portion of the screened interval of the well or other interval selected by the field team leader. If turbidity and its impact on metals analyses are a concern, a period of time sufficient should be allowed to mitigate effects of pump or tubing placement. After it has been determined that sampling may proceed, the pump is turned on and operated at a rate that does not cause significant drawdown of the water column, as measured using a water level sounder. During sampling, sufficient water to supply enough volume for the analytes of concern and the purge parameters is pumped. Purging should continue until purge parameters stabilize, generally three consecutive stable sets of readings, before samples are collected.

4.5.2 HydraSleeves™

HydraSleeves™ are grab sampling devices that are deployed in a closed configuration then opened in the desired interval for sample collection. The following is a summary of its operation:

1. Sampler placement - Reusable weight is attached and the HydraSleeve™ is lowered and placed at the desired position in the well screen. In-situ water pressure keeps the reed valve closed, preventing water from entering the sampler. Well is allowed to return to equilibrium.

2. Sample collection - The reed valve opens to allow filling when the sampler is moved upward faster than 1 foot per second, either in one continuous upward pull or by cycling the sampler up and down to sample a shorter interval. There is no change in water level, and only minimal agitation during collection.
3. Sample retrieval - When the flexible sleeve is full, the reed valve closes and the sampler can be recovered without entry of extraneous overlying fluids. Samples are removed by puncturing the sleeve with the pointed discharge tube and draining the contents into containers for sampling or field measurement.

4.5.3 Passive Diffusion Bags

Passive diffusion bag (PDB) samplers are bags comprised of low-density polyethylene (LDPE) plastic and containing analyte-free water, preferably with no headspace. The bags are deployed, with stainless steel weights, to the desired sample interval and are allowed to equilibrate with the water at the point of deployment in the well. A deployment period of a minimum of 14 days is recommended to ensure equilibration prior to removal.

After 14 days, the bags are opened with a puncture device or other cutting implement and the contents transferred to containers for sampling or field measurement.

4.5.4 General Considerations for Micro-Purge or No-Purge Sampling

When using the Micro-Purge method, it may be advisable to deploy the tubing or pump in advance of sample collection. Introducing the tubing or pump into the screened interval is likely to dislodge sediment and other fines that have settled or bridged on the well screen material and the gravel pack media behind the screen. If sampling is conducted immediately, turbidity issues may render this method impractical from a parameter stability standpoint.

HydraSleeves™ and PDBs must be evaluated for appropriateness for analytes of concern.

4.6 Sample Preservation

After sample collection, all samples requiring preservation must be preserved as soon as practical. Consult the ASBLOQAM for the correct preservative for the particular analytes of interest. All samples preserved using a pH adjustment (except VOCs) must be checked, using pH strips, to ensure that they were adequately preserved. This is done by pouring a small volume of sample over the strip. Do not place the strip in the sample. Samples requiring reduced temperature storage should be placed on ice immediately.

4.7 Special Sample Collection Procedures

4.7.1 Trace Organic Compounds and Metals

Special sample handling procedures should be instituted when trace contaminant samples are being collected. All sampling equipment, including pumps, bailers, water level measurement equipment, etc., which comes into contact with the water in the well must be cleaned in accordance with the cleaning procedures described in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206). Pumps should not be used for sampling unless the interior and exterior portions of the pump and the discharge hoses are thoroughly cleaned. Blank samples should be collected to determine the adequacy of cleaning prior to collection of any sample using a pump other than a peristaltic pump.

4.7.2 Order of Sampling with Respect to Analytes

In many situations when sampling permanent or temporary monitoring wells, an adequate purge, with respect to turbidity, is often difficult to achieve. Removal and insertion of equipment after the purge and prior to actual sampling may negate the low turbidities achieved during purging and elevate turbidity back to unacceptable levels. For this reason, it is important that special efforts be used to minimize any disturbance of the water column after purging and to collect the aliquot for metals first. Therefore, the preferred order of sampling is metals first, followed by other inorganic analytes, extractable organic compounds and volatile organic compounds.

4.7.3 Filtering

As a standard practice, ground water samples will not be filtered for routine analysis. Filtering will usually only be performed to determine the fraction of major ions and trace metals passing the filter and used for flow system analysis and for the purpose of geochemical speciation modeling. Filtration is not allowed to correct for improperly designed or constructed monitoring wells, inappropriate sampling methods, or poor sampling technique.

When samples are collected for routine analyses and are filtered, both filtered and non-filtered samples will be submitted for analyses. Samples for organic compounds analysis should not be filtered. Prior to filtration of the ground water sample for any reason other than geochemical speciation modeling, the following criteria must be demonstrated to justify the use of filtered samples for inorganic analysis:

1. The monitoring wells, whether temporary or permanent, have been constructed and developed in accordance with the SESD Guidance Document, Design and Installation of Monitoring Wells (SESDGUID-001).

2. The ground water samples were collected using sampling techniques in accordance with this section, and the ground water samples were analyzed in accordance with USEPA approved methods.
3. Efforts have been undertaken to minimize any persistent sample turbidity problems. These efforts may consist of the following:
 - Redevelopment or re-installation of permanent ground water monitoring wells.
 - Implementation of low flow/low stress purging and sampling techniques.
4. Turbidity measurements should be taken during purging and sampling to demonstrate stabilization or lack thereof. These measurements should be documented in the field notes. If the ground water sample appears to have either a chemically-induced elevated turbidity, such as would occur with precipitate formation, or a naturally elevated colloid or fine, particulate-related turbidity, filtration will not be allowed.

If filtration is necessary for purposes of geochemical modeling or other **pre-approved** cases, the following procedures are suggested:

1. Accomplish in-line filtration through the use of disposable, high capacity filter cartridges (barrel-type) or membrane filters in an in-line filter apparatus. The high capacity, barrel-type filter is preferred due to the higher surface area associated with this configuration. If a membrane filter is utilized, a minimum diameter of 142 mm is suggested.
2. Use a 5 μm pore-size filter for the purpose of determining the colloidal constituent concentrations. A 0.1 μm pore-size filter should be used to remove most non-dissolved particles.
3. Rinse the cartridge or barrel-type filter with 500 milliliters of the solute (groundwater to be sampled) prior to collection of sample. If a membrane filter is used, rinse with 100 milliliters of solute prior to sample collection.

Potential differences could result from variations in filtration procedures used to process water samples for the determination of trace element concentrations. A number of factors associated with filtration can substantially alter "dissolved" trace element concentrations; these include filter pore size, filter type, filter diameter, filtration method, volume of sample processed, suspended sediment concentration, suspended sediment grain-size distribution, concentration of colloids and colloiddally-associated trace elements, and concentration of organic matter. Therefore, consistency is critical in the comparison of short-term and long-term results. Further guidance on filtration may be obtained from the following: 1) Metals in Ground Water: Sampling Artifacts and Reproducibility; 2) Filtration of Ground Water Samples for Metals Analysis; and 3) Ground Water Sampling - A Workshop Summary. See Section 1.4, References, for complete citation for these documents.

Bacterial Sampling

Whenever wells (normally potable wells) are sampled for bacteriological parameters, care must be taken to ensure the sterility of all sampling equipment and all other equipment entering the well. Further information regarding bacteriological sampling is available in the following: 1) Sampling for Organic Chemicals and Microorganisms in the Subsurface; 2) Handbook for Evaluating Water Bacteriological Laboratories; and 3) Microbiological Methods for Monitoring the Environment, Water and Wastes. See Section 1.4, References, for complete citation for these documents.

4.8 Specific Sampling Equipment Quality Assurance Techniques

All equipment used to collect ground water samples shall be cleaned as outlined in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206) and repaired, if necessary, before being stored at the conclusion of field studies. Cleaning procedures utilized in the field or field repairs shall be thoroughly documented in field records.

4.9 Auxiliary Data Collection

During ground water sample collection, it is important to record a variety of ground water related data. Included in the category of auxiliary data are water levels measured according to the SESD Operating Procedure for Groundwater Level and Well Depth Measurement (SESDPROC-105), well volume determinations (Section 3.1.1, Purging and Purge Adequacy), pumping rates during purging (see below), and occasionally, drillers or boring logs. This information should be documented in the field records.

4.9.1 Well Pumping Rate – Bucket/Stop Watch Method

The pumping rate for a pump can be determined by collecting the discharge from the pump in a bucket of known volume and timing how long it takes to fill the bucket. The pumping rate should be in gallons per minute. This method shall be used primarily with pumps with a constant pump rate, such as gasoline-powered or electric submersible pumps. Care should be taken when using this method with some battery-powered pumps. As the batteries' charge decreases, the pump rate also decreases so that pumping rate calculations using initial, high pump rates may be erroneously high. If this method is used with battery-powered pumps, the rate should be re-checked frequently to ensure accuracy of the pumping rate calculations.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: Surface Water Sampling

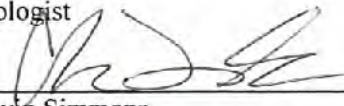
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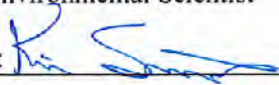
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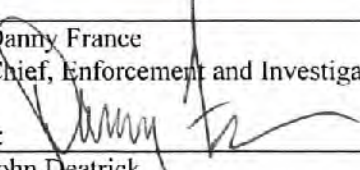
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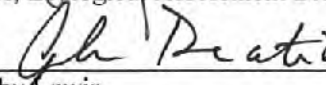
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Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the SESD Document Control Coordinator on the SESD local area network (LAN).

History	Effective Date
SESDPROC-201-R3, <i>Surface Water Sampling</i>, replaces SESDPROC-201-R2. General: Corrected any typographical, grammatical and/or editorial errors. Revision History: Changes were made to reflect the current practice of only including the most recent changes in the revision history. Section 2.3: Item 4 was revised to reflect practice of using individual single-use preservative vials instead of preservatives prepared by ASB.	February 28, 2013
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SESDPROC-201-R0, <i>Surface Water Sampling</i>, Original Issue	February 05, 2007

TABLE OF CONTENTS

1	General Information	4
1.1	Purpose	4
1.2	Scope/Application	4
1.3	Documentation/Verification.....	4
1.4	References.....	4
1.5	General Precautions	6
1.5.1	<i>Safety</i>	6
1.5.2	<i>Procedural Precautions</i>	6
2	Special Sampling Considerations	7
2.1	Volatile Organic Compounds (VOC) Analysis	7
2.2	Special Precautions for Surface Water Sampling.....	7
2.3	Sample Handling and Preservation Requirements.....	8
2.4	Quality Control	9
2.5	Records	9
3	General Considerations.....	10
3.1	General.....	10
3.2	Equipment Selection Considerations	10
4	Dipping Using Sample Container	11
5	Scoops.....	12
6	Peristaltic Pumps	13
7	Discrete Depth Samplers	14
8	Bailers	15
9	Buckets.....	16
10	Submersible Pumps	17
11	Automatic Samplers	18
12	Trace-Level Mercury Sampling	19

1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when collecting surface water samples for field screening or laboratory analysis.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when collecting and handling surface water samples in the field. On the occasion that SESD field personnel determine that any of the procedures described in this section are either inappropriate, inadequate or impractical and that another procedure must be used to obtain a surface water sample, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version

SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version

SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

SESD Operating Procedure for Field pH Measurement, SESDPROC-100, Most Recent Version

SESD Operating Procedure for Field Specific Conductance Measurement, SESDPROC-101, Most Recent Version

SESD Operating Procedure for Field Turbidity Measurement, SESDPROC-103, Most Recent Version

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, SESDPROC-206, Most Recent Version

SESD Operating Procedure for Packaging, Marking, Labeling and Shipping of Environmental and Waste Samples, SESDPROC-209, Most Recent Version

Title 49 Code of Federal Regulations, Pts. 171 to 179, Most Recent Version

United States Environmental Protection Agency (US EPA). 1981. "Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples," Memo from David Weitzman, Work Group Chairman, Office of Occupational Health and Safety (PM-273), April 13, 1981.

US EPA. 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Analytical Support Branch Laboratory Operations and Quality Assurance Manual. Region 4 SEDS, Athens, GA, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SEDS, Athens, GA, Most Recent Version

SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when collecting surface water samples. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines should be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when collecting surface water samples.

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party.
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.
- Documentation of field sampling is done in a bound logbook.
- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- All shipping documents, such as air bills, bills of lading, etc., shall be retained by the project leader and stored in a secure place.

2 Special Sampling Considerations

2.1 Volatile Organic Compounds (VOC) Analysis

Surface water samples for VOC analysis must be collected in 40 ml glass vials with Teflon® septa. The vial may be either preserved with concentrated hydrochloric acid or they may be unpreserved. Preserved samples have a two-week holding time, whereas, unpreserved samples have only a seven-day holding time. In the great majority of cases, the preserved vials are used to take advantage of the extended holding time. In some situations, however, it may be necessary to use the unpreserved vials. For example, if the surface water sample contains a high concentration of dissolved calcium carbonate, there may be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles. This will render the sample unacceptable. In this case, unpreserved vials should be used and arrangements must be confirmed with the laboratory to ensure that they can accept the unpreserved vials and meet the shorter sample holding times.

The samples should be collected with as little agitation or disturbance as possible. The vial should be filled so that there is a reverse or convex meniscus at the top of the vial and absolutely no bubbles or headspace should be present in the vial after it is capped. After the cap is securely tightened, the vial should be inverted and tapped on the palm of one hand to see if any undetected bubbles are dislodged. If a bubble or bubbles are present, the vial should be topped off using a minimal amount of sample to re-establish the meniscus. Care should be taken not to flush any preservative out of the vial during topping off. If, after topping off and capping the vial, bubbles are still present, a new vial should be obtained and the sample re-collected.

Samples for VOC analysis must be collected using either stainless steel or Teflon® equipment.

2.2 Special Precautions for Surface Water Sampling

- A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- Sample containers for samples suspected of containing high concentrations of contaminants shall be stored separately.
- All background or control samples shall be collected and placed in separate ice chests or shipping containers. Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area. Samples of waste or highly contaminated media must not be

placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.

- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other members collect the samples.
- Samplers must use new, verified and certified-clean disposable or non-disposable equipment cleaned according to procedures contained in SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, or SESD Operating Procedure for Field Cleaning and Decontamination at the FEC, SESDPROC-206, for collection of samples for trace metals or organic compound analyses.

2.3 Sample Handling and Preservation Requirements

1. Surface water samples will typically be collected either by directly filling the container from the surface water body being sampled or by decanting the water from a collection device such as a stainless steel scoop or other device.
2. During sample collection, if transferring the sample from a collection device, make sure that the device does not come in contact with the sample containers.
3. Place the sample into appropriate, labeled containers. Samples collected for VOC analysis must not have any headspace (see Section 2.1, Volatile Organic Compounds (VOC) Analysis). All other sample containers must be filled with an allowance for ullage.
4. All samples requiring preservation must be preserved as soon as practically possible, ideally immediately at the time of sample collection. If preserved VOC vials are used, these will be preserved with concentrated hydrochloric acid by ASB personnel prior to departure for the field investigation. For all other chemical preservatives, SESD will use the appropriate chemical preservative generally stored in an individual single-use vial as described in the SESD Operating Procedure for Field Sampling Quality Control (SESDPROC-011). The adequacy of sample preservation will be checked after the addition of the preservative for all samples, except for the samples collected for VOC analysis. If it is determined that a sample is not adequately preserved, additional preservative should be added to achieve adequate preservation. Preservation requirements for surface water samples are found in the USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM).
5. All samples preserved using a pH adjustment (except VOCs) must be checked, using pH strips, to ensure that they were adequately preserved. This is done by pouring a small volume of sample over the strip. Do not place the strip in the sample. Samples requiring reduced temperature storage should be placed on ice immediately.

2.4 Quality Control

If possible, a control sample should be collected from a location not affected by the possible contaminants of concern and submitted with the other samples. In streams or other bodies of moving water, the control sample should be collected upstream of the sampled area. For impounded bodies of water, particularly small lakes or ponds, it may be difficult or inappropriate to obtain an unbiased control from the same body of water from which the samples are collected. In these cases, it may be appropriate to collect a background sample from a similar impoundment located near the sampled body of water if there is a reasonable certainty that the background location has not been impacted. Equipment blanks should be collected if equipment is field cleaned and re-used on-site or if necessary to document that low-level contaminants were not introduced by pumps, bailers or other sampling equipment.

2.5 Records

Information generated or obtained by SESD personnel will be organized and accounted for in accordance with SESD records management procedures found in SESD Operating Procedure for Control of Records, SESDPROC-002. Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation in accordance with SESD Operating Procedure for Logbooks, SESDPROC-010 and SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005.

3 General Considerations

3.1 General

The surface water sampling techniques and equipment described in the following sections of this procedure are designed to minimize effects on the chemical and physical integrity of the sample. If the procedures in these sections are followed, a representative sample of the surface water should be obtained.

3.2 Equipment Selection Considerations

The physical location of the investigator when collecting a sample may dictate the equipment to be used. If surface water samples are required, direct dipping of the sample container into the stream is desirable. Collecting samples in this manner is possible when sampling from accessible locations such as stream banks or by wading or from low platforms, such as small boats or piers. Wading or streamside sampling from banks, however, may cause the re-suspension of bottom deposits and bias the sample. Wading is acceptable if the stream has a noticeable current (is not impounded), and the samples are collected while facing upstream. If the stream is too deep to wade, or if the sample must be collected from more than one water depth, or if the sample must be collected from an elevated platform (bridge, pier, etc.), supplemental sampling equipment must be used.

To collect a surface water sample from a water body or other surface water conveyance, a variety of methods can be used:

- Dipping Using Sample Container
- Scoops
- Peristaltic Pumps
- Discrete Depth Samplers
- Bailers
- Buckets
- Submersible Pumps
- Automatic Samplers

Regardless of the method used, precautions should be taken to ensure that the sample collected is representative of the water body or conveyance. These methods are discussed in the following sections.

4 Dipping Using Sample Container

A sample may be collected directly into the sample container when the surface water source is accessible by wading or other means. The sampler should face upstream if there is a current and collect the sample without disturbing the bottom sediment. The surface water sample should always be collected prior to the collection of a sediment sample at the same location. The sampler should be careful not to displace the preservative from a pre-preserved sample container, such as the 40-ml VOC vial.

5 Scoops

Stainless steel scoops provide a means of collecting surface water samples from surface water bodies that are too deep to access by wading. They have a limited reach of about eight feet and, if samples from distances too far to access using this method are needed, a mobile platform, such as a boat, may be required.

Stainless steel scoops are useful for reaching out into a body of water to collect a surface water sample. The scoop may be used directly to collect and transfer a surface water sample to the sample container, or it may be attached to an extension in order to access the selected sampling location.

6 Peristaltic Pumps

Another device that can be effectively used to sample a water column, such as a shallow pond or stream, is the peristaltic pump/vacuum jug system. The peristaltic pump can be used to collect a water sample from any depth if the pump is located at or near the surface water elevation. There is no suction limit for these applications. The use of a metal conduit to which the tubing is attached, allows for the collection of a vertical sample (to about a 25-foot depth) which is representative of the water column. The tubing intake is positioned in the water column at the desired depth by means of the conduit. Using this method, discrete samples may be collected by positioning the tubing intake at one depth or a vertical composite may be collected by moving the tubing intake at a constant rate vertically up and down the water column over the interval to be composited.

Samples for VOC analysis cannot be collected directly from the peristaltic pump discharge or from the vacuum jug. If a peristaltic pump is used for sample collection and VOC analysis is required, the VOC sample must be collected using one of the “soda straw” variations. Ideally, the tubing intake will be placed at the depth from which the sample is to be collected and the pump will be run for several minutes to fill the tubing with water representative of that interval. After several minutes, the pump is turned off and the tubing string is retrieved. The pump speed is then reduced to a slow pumping rate and the pump direction is reversed. After turning the pump back on, the sample stream is collected into the VOC vials as it is pushed from the tubing by the pump. Care must be taken to prevent any water that was in contact with the silastic pump head tubing from being incorporated into the sample.

7 Discrete Depth Samplers

When discrete samples are desired from a specific depth, and the parameters to be measured do not require a Teflon®-coated sampler, a standard Kemmerer or Van Dorn sampler may be used. The Kemmerer sampler is a brass cylinder with rubber stoppers that leave the ends of the sampler open while being lowered in a vertical position, thus allowing free passage of water through the cylinder. The Van Dorn sampler is plastic and is lowered in a horizontal position. In each case, a messenger is sent down a rope when the sampler is at the designated depth, to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill respective sample containers. With a rubber tube attached to the valve, dissolved oxygen sample bottles can be properly filled by allowing an overflow of the water being collected. With multiple depth samples, care should be taken not to disturb the bottom sediment, thus biasing the sample.

When metals and organic compounds parameters are of concern, then a double-check valve, stainless steel bailer or Kemmerer sampler should be used to collect the sample.

8 Bailers

Teflon® bailers may also be used for surface water sampling if the study objectives do not necessitate a sample from a discrete interval in the water column. A closed-top bailer with a bottom check-valve is sufficient for many studies. As the bailer is lowered through the water column, water is continually displaced through the bailer until the desired depth is reached, at which point the bailer is retrieved. This technique may not be successful where strong currents are found.

9 Buckets

A plastic bucket can be used to collect samples for measurement of water quality parameters such as pH, temperature, and conductivity. Samples collected for analysis of classical water quality parameters including but not limited to ammonia, nitrate-nitrite, phosphorus, and total organic carbon may also be collected with a bucket. Typically, a bucket is used to collect a sample when the water depth is too great for wading, it is not possible to deploy a boat, or access is not possible (excessive vegetation or steep embankments) and the water column is well mixed. The water body is usually accessed from a bridge. The bucket is normally lowered by rope over the side of the bridge. Upon retrieval, the water is poured into the appropriate sample containers.

Caution should be exercised whenever working from a bridge. Appropriate measures should be taken to insure the safety of sampling personnel from traffic hazards.

10 Submersible Pumps

Submersible pumps can be used to collect surface water samples directly into a sample container. The constituents of interest should be taken into consideration when choosing the type of submersible pump and tubing to be used. If trace contaminant sampling of extractable organic compounds and/or inorganic analytes will be conducted, the submersible pump and all of its components should be constructed of inert materials such as stainless steel and Teflon®. The tubing should also be constructed of Teflon®. If re-using the same pump between sample locations, the pump should be decontaminated using SESD Operating Procedure for Field Equipment Cleaning and Decontamination, (SESDPROC-205). New tubing should be used at each sample location.

If the samples will be analyzed for classical parameters such as ammonia, nitrate-nitrite, phosphorus, or total organic carbon, the pump and tubing may be constructed of components other than stainless steel and Teflon®. The same pump and tubing may be re-used at each sampling station after rinsing with deionized water and then purging several volumes of sample water through the pump and tubing prior to filling the sample containers.

Either a grab or composite sample can be collected using a submersible pump. A composite sample can be collected by raising and lowering the pump throughout the water column. If a composite sample is collected, it may be necessary to pump the sample into a compositing vessel for mixing prior to dispensing into the sample containers. If a compositing vessel is required, it should be constructed of materials compatible with the constituents of concern and decontaminated between sample stations according to appropriate procedures, again depending on the constituents of concern.

11 Automatic Samplers

Where unattended sampling is required (e.g., storm-event sampling, time-of-travel studies) an automatic sampler may be used. The automatic sampling device may be used to collect grab samples based on time, in-stream flow or water level or used to collect composite samples as dictated by the study data needs. The automatic sampling device should be calibrated prior to deployment to insure the proper volume is collected. The manufacturer's instruction manual should be consulted for automatic sampler operation.

12 Trace-Level Mercury Sampling

In order to prevent contamination during sample collection, Region 4 has developed this sampling procedure for trace-level mercury analysis (< 1 part per trillion). This procedure is based on EPA Method 1669.

A vacuum chamber assembly is utilized to collect surface water samples for trace-level mercury analyses. The vacuum chamber assembly consists of the following: 1) an airtight acrylic, cylindrical chamber with an o-ring sealed lid to hold the sample bottle, 2) a Teflon® sample tubing that connects to a centered Teflon® compression fitting on top of the chamber. The other end of the tubing passes through a rigid Teflon® pole for stability and has a modified magnetic screen holder at the intake, and a hand vacuum pump. The chamber is designed to hold a 2-liter sample bottle; however, smaller sample containers may be utilized with a spacer inserted into the chamber. A two inch square of 100 µm Nitex® screen is used on the magnetic screen holder at the intake to prevent large pieces of debris from entering the sample. The screen does not prevent the passage of particulate organic matter which is often prevalent in surface water. The vacuum chamber has a second off-center compression fitting with a 4 inch piece of Teflon® tubing inserted in the fitting. A piece of clear Tygon® tubing approximately 18-24 inches long is placed over the small piece of Teflon®. The Teflon® adds stability to the tubing and keeps it from crimping. The Tygon® is attached to the hand pump and the chamber with electrical tape. The Nitex® screen intake is inserted into the water to be sampled and a vacuum is pulled on the chamber by means of the hand vacuum pump, thus drawing a water sample into a sample container placed directly beneath the intake tubing within the chamber.

Teflon® bottles or 300-Series glass bottles with single use Teflon®-lined caps may be used for sample collection. All sample containers used for collection of trace-level mercury water samples must be pre-cleaned in a laboratory as described in EPA Method 1631. Teflon® containers should also be etched on the outside of the bottle with a unique identification number for QA purposes. All bottles for trace-level sampling must be double bagged in re-sealable bags. Water samples collected for total, inorganic, methyl or ethyl mercury analyses are pumped into appropriately cleaned bottles. Preservation should be done in a clean room laboratory that has been specifically prepared for the preparation of trace level samples (positive pressure ventilation, sticky floor mats, etc.). Preservation must occur within 48 hours of sample collection, sooner if possible. Region 4 utilizes laboratory preservation of trace-level mercury samples in order to minimize the potential for contamination, and if split samples are required, they must be split in a trace-level clean room laboratory.

The following quality assurance/quality control (QA/QC) samples are collected in conjunction with low-level mercury samples:

- bottle blanks
- equipment blanks

- air deposition blanks
- trip blanks
- duplicates and
- splits

A bottle blank is prepared in the lab with reagent-grade water to ensure the cleanliness of the bottles prior to use in the field. After decontamination of the Teflon® tubing by pumping and discarding several sample container volumes of reagent-grade water through the tubing, (using the same amount of water used for sample collection in the field) an equipment blank sample is collected into an appropriately pre-cleaned sample container. Equipment blanks are collected at the beginning of each field trip and at the end of each day. The bottle blank and the equipment blank do not go out into the field and are preserved at the end of the day with the regular field samples.

Air deposition blanks are collected to determine if airborne mercury is present at the time of sample collection. The air deposition blanks consist of a pre-cleaned mercury sample container, filled with reagent-grade water by the laboratory that prepared the containers, and is shipped with the containers to the field. The air deposition blank is uncapped using “clean hands”/ “dirty hands” procedures (see below) and set near the sampling location throughout the duration of the mercury sample collection for that particular station. Once the mercury sample is collected, the air deposition blank is recapped and handled and processed with the other mercury samples. One air deposition blank is collected each day by each field crew unless atmospheric conditions or site conditions warrant additional blanks.

Trip blanks are utilized to determine if any contaminants of interest to the study are potentially introduced to the samples during storage and transport to the laboratory. Trip blanks are prepared by the laboratory which supplies the mercury sample containers. The trip blanks consist of cleaned bottles which are filled with reagent-grade water by the laboratory and shipped with the other clean sample containers. A dark plastic bag is placed in each cooler that will hold the trace-level water samples. One trip blank is placed in each trace-level cooler of samples and returned to the laboratory with the ambient trace-level water samples. All trace-level samples should be kept in the dark until they are preserved. The trip blanks are never opened in the field. Trip blanks are preserved in the clean room.

Duplicate samples are discrete samples collected at the same site and time to measure variability of collected samples and to assess sample collection consistency. Sample splits are aliquots of a minimum 500 ml poured from a single ambient sample. They must be split in a trace-level clean room laboratory.

In order to prevent cross contamination in samples analyzed for trace-level mercury in ambient surface waters, clean sampling protocols must be employed throughout the sampling effort. For each sampling event, one sampling team member is designated as “clean hands” and one as “dirty hands” (see below). All operations involving contact

with the sample bottle and transfer of the sample from the sample collection device to the sample bottle are handled by the individual designated as “clean hands.” “Dirty hands” is responsible for preparation of the sampling device (except the sample container) and for all other activities that do not involve direct contact with the sample.

Prior to sample collection with the vacuum chamber assembly, the Teflon® line is cleaned at each station by rinsing with ambient water as follows: A 2-liter poly bottle is placed into the chamber and filled half full with ambient water. The bottle is swirled to rinse it and the water is discarded downstream of sampling area. The same 2-liter poly bottle can be used at each station. Additional cleaning measures are not recommended as long as the chamber assembly is only used to collect ambient surface water samples. Detergent washes and acid rinses are not conducted due to potential mercury contamination from these solutions. If applicable, samples for other analyses can be collected in a poly bottle with the vacuum chamber assembly but should be collected before the trace-level sample as an additional means of flushing the sampling line prior to collection of the trace-level samples. It is not necessary to implement the “clean hands”/ “dirty hands” method for collection of non-mercury samples, but latex or vinyl gloves should be worn when any samples are collected.

Following are procedures for cleaning the vacuum chamber tubing and collection of ancillary water quality samples, if applicable:

1. Carefully approach the sampling station from downstream and downwind if possible.
2. While wearing latex or vinyl gloves, place an uncapped 2-liter poly bottle into the chamber and secure the chamber lid by attaching the spring-loaded clamps.
3. Place a new square of 100 µm Nitex® screen in the magnetic screen holder. Place the intake beneath the surface of the water (mid-depth or six inches, whichever is less) and hold firmly in place. Care should be taken not to disturb sediment particles in very shallow waters (< 4 inches deep).
4. Squeeze the hand pump until liquid starts to fill the bottle in the chamber. When the bottle is approximately half full, release the vacuum on the chamber, remove the bottle, swirl the contents and discard the water downstream. Repeat this rinse. If ancillary water quality samples are to be collected, return the 2-liter poly bottle to the chamber and pump the required volume of water to fill the appropriate ancillary sample containers. Remove the 2-liter bottle from the chamber and cap. Fill the ancillary sample bottles upon completion of the mercury sample collection.

Water samples for trace level mercury analyses should be collected immediately after the ancillary water samples have been collected according to the following procedures:

1. “Clean hands” should put on a pair of latex or vinyl gloves, then a pair of shoulder length polyethylene gloves.
2. “Dirty hands” should put on a pair of latex or vinyl gloves, retrieve the double bagged trace level sample bottle from the cooler, and open the outer bag. “Clean hands” should open the inner bag and remove the pre-cleaned Teflon® or glass bottle.
3. “Dirty hands” should open the lid on the chamber. “Clean hands” should place the sample bottle in the chamber, remove the bottle top and place it inside the chamber with the bottle.
4. “Dirty hands” should close and secure the chamber lid and using the hand pump, fill the container. The sample container should be filled to overflowing. “Dirty hands” should then release the vacuum and open the lid on the chamber.
5. “Clean hands” should place the top on the sample bottle, remove it from the chamber and place it in the inner bag and seal the bag. “Dirty hands” should seal the outer bag and place the sample in the black bag in the dark cooler. Only coolers dedicated to storage and transport of trace-level mercury samples should be used.

Standard Operating Procedure SOP-SM-9
Gas Stripping Cell Instructions
Microseeps, Inc.



Gas Stripping Cell Instructions

Technical Guidance from Microseeps

Page 1 of 2

Sampling
Questions?

Call
800-659-2887
Mon. - Fri.
9 -5 EST

Installation and Operation

To place the gas stripping cell into service:

1. Remove one of the cell assemblies from the packing carton. Refer to Figure 1 (following pages) to become familiar with the parts of the cell.
2. Connect the inlet tube of the cell to the outlet of your pump. The inlet tube is designed to connect to ¼ inch O.D. hard tubing. Secure the connection (nylon wire ties are recommended).
3. Insert the drain tube of the cell into a waste container, keeping the end of the tube at the bottom of the container. Any waste container of suitable size may be used. A 2-liter soda pop bottle may be placed in the waste container to determine pumping flow rate.
4. Secure the cell assembly so that the housing cover is above the glass housing (i.e. upright). A ring stand and clamp are recommended for this purpose.
5. Turn the pump on and check for leaks. If any leaks are found seal them before proceeding. Measure, in ml per minute, the flow rate of the pump. If a 2-liter soda pop bottle was used, the flow rate can be determined by measuring how many minutes it takes to fill the bottle, then substituting the measured time into the following equation.

Flow = 2000 ml/time to fill
in minutes

Consult table to determine
the equilibrium time
needed to gas strip at this
flow rate.

Flow rate (ml/min)	Sampling time (min)
100-120	30
130-150	25
160-200	20
210-300	15
>300	10

NOTE: Use a flow rate between 100 ml/min and 350 ml/min. Do not turn off the pump.

6. Unclamp the cell assembly, invert it and re-secure the assembly in the inverted position. Make sure the drain tube is still in the waste container and the end of the drain tube is near the bottom of the bottle.
 7. Connect the (supplied) stopcock to the syringe and the (supplied) needle to the stopcock. Place the stopcock in the open position (i.e. so that the stopcock handle is in-line with the syringe). Draw the plunger back on the syringe to the 20.0-mL mark. Keeping the cell in the inverted position, insert the needle into the needle guide. Pierce the septum and inject the air into the cell. Then remove the needle and syringe from the assembly and carefully cover the needle. Do not discard the syringe apparatus.
 8. Start timing and let the ground water pump through the cell for the time specified in Table 1 for your particular pumping speed. Meanwhile, be sure that the sample vial is properly labeled and that the flow rate and any other relevant field data are recorded in the field log.
- NOTE: Be sure to keep the end of the drain tube at the bottom of the waste container. This will insure that outside air is not drawn into the cell. Failure to do this will invalidate the sample.
9. When the equilibration time is up, turn off the pump, unclamp the cell and re-clamp it in its upright position. Verify that the plunger of the syringe is pushed all the way in and that the stopcock is in the open position, then insert the needle into the needle guide and pierce the septum. Withdraw 1-mL of gas by pulling back on the syringe plunger while holding the syringe body in place, remove the syringe from the cell and expel the sample. Immediately re-insert the needle into the needle guide and pierce the septum. Withdraw a 15-mL sample of gas and, with the needle still through the septum, close the stopcock. Rapidly withdraw the needle from the septum and place it through septum on the sample vial (see Figure 2, next page). Open the stopcock and completely depress the syringe barrel. With the syringe barrel completely depressed, separate the sample vial and the syringe with a quick pull. Discard the syringe apparatus according to Local, State and Federal regulations.





Gas Stripping Cell Instructions

Technical Guidance from Microseeps

Page 2 of 2

Sampling
Questions?

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Decontamination/Cleaning

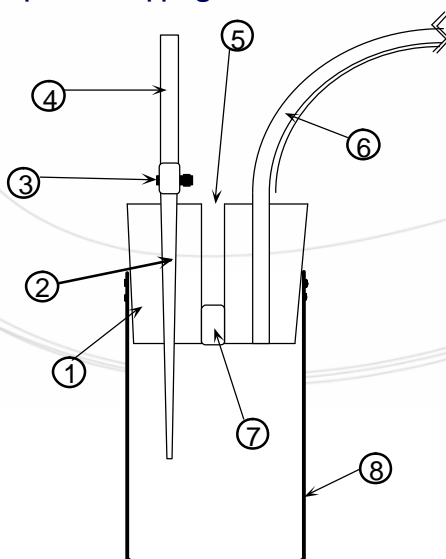
Pump at least 1 liter of potable water through the cell.
The cell assembly is now ready for re-use.

The only expendable part of the cell is the sampling septum (part 7). Normally, each septum may be used for the collection of 5-10 samples. If bubbles are seen rising up from the septum when the cell is inverted the septum **MUST** be replaced.

Figure 1.

Cross section of Microseeps Gas Stripping Cell

1. Housing Cover
2. Jet Spray Nozzle
3. Nylon Tie
4. Inlet Tube
5. Needle Guide Port
6. Drain Tube
7. Replaceable Septum
8. Glass Housing



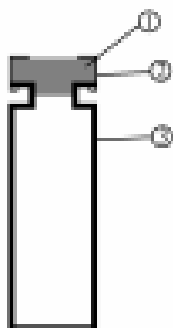
Replacing the Sampling Port Septum

All part numbers refer to Figure 1.

1. Remove the housing cover (part 1) from the glass housing (part 8).
2. Use a handy, blunt tipped object to push the replaceable septum (part 7) out of the housing cover. The cover to a needle works well for this purpose, but be sure that the needle is **NOT** in the cover. Discard the old septum.
3. Take a new septum and wet both the new septum and the housing cover with potable water.
4. Carefully using the same blunt instrument used in step three above, slide the new septum into the hole from which the old septum was removed. The bottom of the new septum must be flush with the narrow end of the housing cover.
5. If the housing cover is not still wet, wet it again with potable water. Place the bottom end of the housing cover into the glass housing and push it in until less than 3/8" are above the rim of the glass housing. This may require some force.
6. Follow the cleaning procedures described above to prepare the cell for a return to service.

Figure 2.

Cross section of septum bottle



1. Septum
2. Metal Closure
3. Glass vial



Microseeps Gas Stripping Cell Instructions

[Back to Microseeps Homepage](#)

[Choosing Sampling Pumps for Hydrogen from Deep Wells](#)

INSTALLATION AND OPERATION

To place the gas stripping cell into service:

Image 1.



[Back to step 11](#)

1. Remove one of the cell assemblies from the packing carton. See [Figure 1](#).

2. **Image 1.** Connect the inlet tube of the cell to the outlet of your pump. The inlet tube is designed to connect to 1/4 O.D. hard tubing. Secure the connection using binder clips or cable ties.

3. Insert the drain tube of the cell into a waste container, keeping the end of the tube at the bottom of the container. Any waste container of suitable size may be used. A 2-Liter soda pop bottle may be placed in the waste container to determine pumping flow rate.

4. Secure the cell assembly so that the housing cover (stopper) is above the glass housing (i.e. upright). A ring stand and clamp are recommended for this purpose.

5. Turn the pump on and check for leaks. If any leaks are found, seal them before proceeding.

Image 2.



6. **Image 2.** Measure, in mL per minute, the flow rate of the pump. If a 2-Liter soda pop bottle is used, the flow rate can be determined by measuring how many minutes it takes to fill the bottle and substituting the measured time into the following equation:

Flow = 2000 mL/Time to fill (in minutes)

Consult [Table 1](#) to determine the equilibrium time needed to bubble strip at this flow rate.

Note: Use a flow rate between 100 mL/min. and 500 mL/min. **Do not turn off the pump.**

Table 1.

<u>Flow rate (ml/ min)</u>	<u>Sampling time (min)</u>
100-120	30
130-150	25
160-200	20
210-300	15
>300	10

[Return to Step 6](#)

[Return to Step 10](#)

Image 3.



7. **Image 3.** Unclamp the cell assembly, invert it, and re-secure the assembly in the inverted position. Make sure the drain tube is still in the waste container and the end of the drain tube is near the bottom of the bottle.

Image 4.



8. **Image 4.** Connect the stopcock to the syringe and the needle to the stopcock (zoom in on image). Place the stopcock in the open position (so that the stopcock handle is in-line with the syringe). Draw the plunger back on the syringe to the 20.0 mL mark pulling ambient air into the syringe.

Image 5.



9. **Image 5.** Keeping the cell in the inverted position, insert the needle into the needle guide. Pierce the septum and inject the air into the cell creating the bubble. Withdraw the needle from the assembly and carefully place the needle into the cover. Do not discard the syringe apparatus.

10. Start timing and let the groundwater pump through the cell for time specified in [Table 1](#) for your particular pumping speed. Meanwhile, be sure that the sample vial is properly labeled and that the flow rate and any other relevant field data are recorded in the field log.

Note: Be sure to keep the end of the drain tube at the bottom of the waste container. This will insure that outside air is not drawn into the cell. **Failure to do this will invalidate the sample.**

11. When equilibration time is up, **turn off the pump**, unclamp the cell, and re-clamp it in its upright position. See [Image 1](#). Verify that the plunger of the syringe is pushed all the way in and that the stopcock is in the open position.

Image 6.



12. **Image 6.** Insert the needle into the needle guide and pierce the septum. Withdraw 1 mL of gas by pulling back on the syringe plunger while holding the syringe body in place. Remove the syringe from the cell and expel the sample.

13. Immediately re-insert the needle into the needle guide and pierce the septum. Withdraw a 15 mL sample of gas (being careful not to pull any water into the syringe). With the needle still through the septum, close the stopcock and withdraw the needle from the septum.

Image 7.



14. **Image 7.** Immediately insert the needle through the septum on the sample vial. Keeping the syringe and vial "in line", open the stopcock and completely depress the syringe plunger injecting the entire sample into the vial.

Image 8.



15. **Image 8.** Keeping the plunger depressed, quickly remove the vial from the needle. Your sample is now ready to be packaged and shipped back to Microseeps for analysis. Do not cool the samples.

[back to top](#)

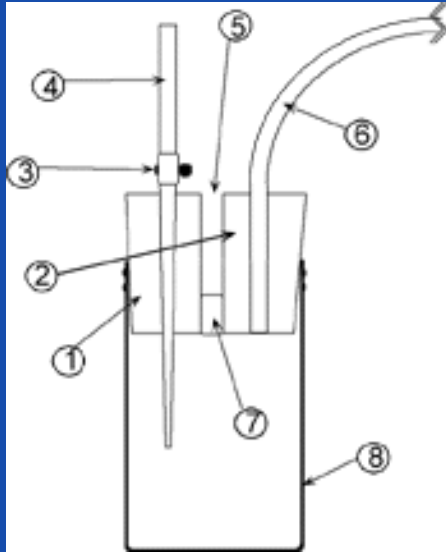
Decontamination/Cleaning

Pump at least 1 liter of potable water through the cell. The cell assembly is now ready for re-use.

The only expendable part of the cell is the sampling septum (part 7). Normally, each septum may be used for the collection of up to 5 samples. If bubbles are seen rising up from the septum when the cell is inverted the septum **MUST be replaced. Instructions for replacing the septum are provided below.**

[back to top](#)

Figure 1. Cross section of Microseeps Gas Stripping Cell



2. Jet Spray Nozzle

3. Nylon Tie

4. Inlet Tube

5. Needle Guide Port

6. Drain Tube

7. Replaceable Septum

8. Glass Housing

[back to top](#)

Replacing the Sampling Port Septum

All part numbers refer to Figure 1.

1. Remove the housing cover (part 1) from the glass housing (part 8).

2. Use a handy, blunt tipped object to push the replaceable septum (part 7) out of the housing cover. The cover to a needle works well for this purpose, but be sure that the needle is **NOT** in the cover. Discard the old septum.

4. Take a new septum and wet both the new septum and the housing cover with potable water.

5. Carefully using the same blunt instrument used in step three above, slide the new septum into the hole from which the old septum was removed. The bottom of the new septum must be flush with the narrow end of the housing cover.

6. If the housing cover is not still wet, wet it again with potable water. Place the bottom end of the housing cover into the glass housing and push it in until less than 3/8" are above the rim of the glass housing. This may require some force.

7. Follow the cleaning procedures described above to prepare the cell for a return to service.

[back to top](#)

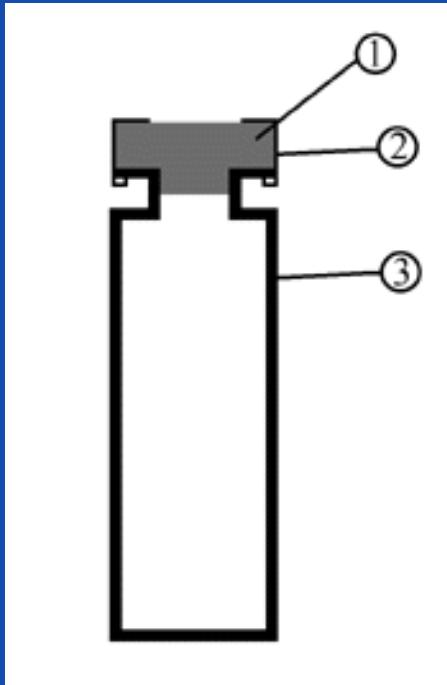


Figure 2. Cross section of septum bottle

1. Septum
2. Metal Closure
3. Glass vial

SAMPLING QUESTIONS?

CALL MICROSEEPS AT **1-412-826-5245**

MON.- FRI. 7:30 AM TO 6 PM EST

[Back to Microseeps Homepage](#)

Appendix C
Laboratory Accreditation Certificates



**LABORATORY
ACCREDITATION
BUREAU**



Certificate of Accreditation

ISO/IEC 17025:2005

Certificate Number L2226

Empirical Laboratories, LLC

621 Mainstream Drive, Suite 270

Nashville TN 37228

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation valid through: November 30, 2015

**R. Douglas Leonard, Jr., President, COO
Laboratory Accreditation Bureau
Presented the 30th of January 2013**

*See the laboratory's Scope of Accreditation for details of accredited parameters

**Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).

Scope of Accreditation For Empirical Laboratories, LLC

621 Mainstream Drive, Suite 270
Nashville, TN 37228
Marcia K. McGinnity
877-345-1113

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.2) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to Empirical Laboratories, LLC to perform the following tests:

Accreditation granted through: November 30, 2015

Testing - Environmental

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B; EPA 624	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B; EPA 624	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	EPA 8260B; EPA 624	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B; EPA 624	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	EPA 8260B; EPA 624	1,1,2-Trichloroethane
GC/MS	EPA 8260B; EPA 624	1,1-Dichloroethane (1,1-DCA)
GC/MS	EPA 8260B; EPA 624	1,1-Dichloroethene (1,1-DCE)
GC/MS	EPA 8260B; EPA 624	1,1-Dichloropropene
GC/MS	EPA 8260B; EPA 624	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B; EPA 624	1,2,3-Trichloropropane
GC/MS	EPA 8260B; EPA 624	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B; EPA 624	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B; EPA 624	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260B; EPA 624	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B; EPA 624	1,2-Dichlorobenzene
GC/MS	EPA 8260B; EPA 624	1,2-Dichloroethane (EDC)
GC/MS	EPA 8260B; EPA 624	1,2-Dichloropropane
GC/MS	EPA 8260B; EPA 624	1,3,5-Trimethylbenzene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B; EPA 624	1,3-Dichlorobenzene
GC/MS	EPA 8260B; EPA 624	1,3-Dichloropropane
GC/MS	EPA 8260B; EPA 624	1,4-Dichlorobenzene
GC/MS	EPA 8260B; EPA 624	1,4-Dioxane
GC/MS	EPA 8260B; EPA 624	1-Chlorohexane
GC/MS	EPA 8260B; EPA 624	2,2-Dichloropropane
GC/MS	EPA 8260B; EPA 624	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	EPA 8260B; EPA 624	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B; EPA 624	2-Chlorotoluene
GC/MS	EPA 8260B; EPA 624	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	EPA 8260B; EPA 624	4-Chlorotoluene
GC/MS	EPA 8260B; EPA 624	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	EPA 8260B; EPA 624	Acetone
GC/MS	EPA 8260B; EPA 624	Acetonitrile
GC/MS	EPA 8260B; EPA 624	Acrolein
GC/MS	EPA 8260B; EPA 624	Acrylonitrile
GC/MS	EPA 8260B; EPA 624	Allyl chloride
GC/MS	EPA 8260B; EPA 624	Benzene
GC/MS	EPA 8260B; EPA 624	Bromobenzene
GC/MS	EPA 8260B; EPA 624	Bromochloromethane
GC/MS	EPA 8260B; EPA 624	Bromodichloromethane
GC/MS	EPA 8260B; EPA 624	Bromoform
GC/MS	EPA 8260B; EPA 624	Bromomethane
GC/MS	EPA 8260B; EPA 624	Carbon Disulfide
GC/MS	EPA 8260B; EPA 624	Carbon Tetrachloride
GC/MS	EPA 8260B; EPA 624	Chlorobenzene
GC/MS	EPA 8260B; EPA 624	Chloroethane
GC/MS	EPA 8260B; EPA 624	Chloroform
GC/MS	EPA 8260B; EPA 624	Chloromethane
GC/MS	EPA 8260B; EPA 624	Chloroprene
GC/MS	EPA 8260B; EPA 624	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	EPA 8260B; EPA 624	cis-1,3-Dichloropropene
GC/MS	EPA 8260B; EPA 624	cis-1,4-Dichloro-2-butene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B; EPA 624	Cyclohexane
GC/MS	EPA 8260B; EPA 624	Dibromochloromethane
GC/MS	EPA 8260B; EPA 624	Dibromomethane
GC/MS	EPA 8260B; EPA 624	Dichlorodifluoromethane (CFC-12)
GC/MS	EPA 8260B; EPA 624	Di-isopropyl ether
GC/MS	EPA 8260B; EPA 624	ETBE
GC/MS	EPA 8260B; EPA 624	Ethyl methacrylate
GC/MS	EPA 8260B; EPA 624	Ethylbenzene
GC/MS	EPA 8260B; EPA 624	Hexachlorobutadiene
GC/MS	EPA 8260B; EPA 624	Hexane
GC/MS	EPA 8260B; EPA 624	Iodomethane
GC/MS	EPA 8260B; EPA 624	Isobutyl alcohol
GC/MS	EPA 8260B; EPA 624	Isopropylbenzene (Cumene)
GC/MS	EPA 8260B; EPA 624	Methacrylonitrile
GC/MS	EPA 8260B; EPA 624	Methyl Acetate
GC/MS	EPA 8260B; EPA 624	Methyl methacrylate
GC/MS	EPA 8260B; EPA 624	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	EPA 8260B; EPA 624	Methylcyclohexane
GC/MS	EPA 8260B; EPA 624	Methylene Chloride, or Dichloromethane
GC/MS	EPA 8260B; EPA 624	Naphthalene
GC/MS	EPA 8260B; EPA 624	n-Butylbenzene
GC/MS	EPA 8260B; EPA 624	n-Propylbenzene
GC/MS	EPA 8260B; EPA 624	p-Isopropyltoluene
GC/MS	EPA 8260B; EPA 624	Propionitrile
GC/MS	EPA 8260B; EPA 624	sec-Butylbenzene
GC/MS	EPA 8260B; EPA 624	Styrene
GC/MS	EPA 8260B; EPA 624	t-Butyl alcohol
GC/MS	EPA 8260B; EPA 624	tert-Amyl methyl ether
GC/MS	EPA 8260B; EPA 624	tert-Butylbenzene
GC/MS	EPA 8260B; EPA 624	Tetrachloroethene (PCE; PERC)
GC/MS	EPA 8260B; EPA 624	Tetrahydrofuran
GC/MS	EPA 8260B; EPA 624	Toluene
GC/MS	EPA 8260B; EPA 624	trans-1,2-Dichloroethene (trans-1,2-DCE)

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B; EPA 624	trans-1,3-Dichloropropene
GC/MS	EPA 8260B; EPA 624	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260B; EPA 624	Trichloroethene (TCE)
GC/MS	EPA 8260B; EPA 624	Trichlorofluoromethane (CFC-11)
GC/MS	EPA 8260B; EPA 624	Vinyl acetate
GC/MS	EPA 8260B; EPA 624	Vinyl Chloride (VC)
GC/MS	EPA 8260B; EPA 624	m,p-Xylenes
GC/MS	EPA 8260B; EPA 624	o-Xylene
GC/MS	EPA 8260B; EPA 624	Xylenes (Total)
GC/MS	EPA 8270C/D; EPA 625	1,1'-Biphenyl
GC/MS	EPA 8270C/D; EPA 625	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D; EPA 625	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C/D; EPA 625	1,2-Dichlorobenzene
GC/MS	EPA 8270C/D; EPA 625	1,2-Diphenylhydrazine
GC/MS	EPA 8270C/D; EPA 625	1,3-Dichlorobenzene
GC/MS	EPA 8270C/D; EPA 625	1,4-Dichlorobenzene
GC/MS	EPA 8270C/D; EPA 625	1,4-Dioxane
GC/MS	EPA 8270C/D; EPA 625	1-Methylnaphthalene
GC/MS	EPA 8270C/D; EPA 625	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D; EPA 625	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D; EPA 625	2,4,6-Trichlorophenol (TCP)
GC/MS	EPA 8270C/D; EPA 625	2,4-Dichlorophenol (DCP)
GC/MS	EPA 8270C/D; EPA 625	2,4-Dimethylphenol
GC/MS	EPA 8270C/D; EPA 625	2,4-Dinitrophenol
GC/MS	EPA 8270C/D; EPA 625	2,4-Dinitrotoluene (DNT)
GC/MS	EPA 8270C/D; EPA 625	2,6-Dichlorophenol
GC/MS	EPA 8270C/D; EPA 625	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D; EPA 625	2-Chloronaphthalene
GC/MS	EPA 8270C/D; EPA 625	2-Chlorophenol
GC/MS	EPA 8270C/D; EPA 625	2-Methylnaphthalene
GC/MS	EPA 8270C/D; EPA 625	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270C/D; EPA 625	2-Nitroaniline
GC/MS	EPA 8270C/D; EPA 625	2-Nitrophenol (ONP)

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C/D; EPA 625	3,3'-Dichlorobenzidine (DCB)
GC/MS	EPA 8270C/D; EPA 625	3-Methylphenol/4-Methylphenol
GC/MS	EPA 8270C/D; EPA 625	3-Nitroaniline
GC/MS	EPA 8270C/D; EPA 625	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	EPA 8270C/D; EPA 625	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D; EPA 625	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D; EPA 625	4-Chloroaniline
GC/MS	EPA 8270C/D; EPA 625	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/D; EPA 625	4-Methylphenol (p-Cresol)
GC/MS	EPA 8270C/D; EPA 625	4-Nitroaniline (PNA)
GC/MS	EPA 8270C/D; EPA 625	4-Nitrophenol (PNP)
GC/MS	EPA 8270C/D; EPA 625	Acenaphthene
GC/MS	EPA 8270C/D; EPA 625	Acenaphthylene
GC/MS	EPA 8270C/D; EPA 625	Acetophenone
GC/MS	EPA 8270C/D; EPA 625	Aniline
GC/MS	EPA 8270C/D; EPA 625	Anthracene
GC/MS	EPA 8270C/D; EPA 625	Atrazine
GC/MS	EPA 8270C/D; EPA 625	Benzaldehyde
GC/MS	EPA 8270C/D; EPA 625	Benzidine
GC/MS	EPA 8270C/D; EPA 625	Benzo(a)anthracene
GC/MS	EPA 8270C/D; EPA 625	Benzo(a)pyrene
GC/MS	EPA 8270C/D; EPA 625	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D; EPA 625	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D; EPA 625	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D; EPA 625	Benzoic Acid
GC/MS	EPA 8270C/D; EPA 625	Benzyl alcohol
GC/MS	EPA 8270C/D; EPA 625	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270C/D; EPA 625	bis(2-Chloroethyl)ether (BCEE)
GC/MS	EPA 8270C/D; EPA 625	bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	EPA 8270C/D; EPA 625	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	EPA 8270C/D; EPA 625	Butyl benzyl phthalate (BBP)
GC/MS	EPA 8270C/D; EPA 625	Caprolactam
GC/MS	EPA 8270C/D; EPA 625	Carbazole

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C/D; EPA 625	Chrysene
GC/MS	EPA 8270C/D; EPA 625	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/D; EPA 625	Dibenzofuran (DBF)
GC/MS	EPA 8270C/D; EPA 625	Diethyl phthalate (DEP)
GC/MS	EPA 8270C/D; EPA 625	Dimethyl phthalate (DMP)
GC/MS	EPA 8270C/D; EPA 625	Di-n-butyl phthalate (DBP)
GC/MS	EPA 8270C/D; EPA 625	Di-n-octyl phthalate (DNOP)
GC/MS	EPA 8270C/D; EPA 625	Fluoranthene
GC/MS	EPA 8270C/D; EPA 625	Fluorene
GC/MS	EPA 8270C/D; EPA 625	Hexachlorobenzene (HCB)
GC/MS	EPA 8270C/D; EPA 625	Hexachlorobutadiene (HCBD)
GC/MS	EPA 8270C/D; EPA 625	Hexachlorocyclopentadiene (HCCPD)
GC/MS	EPA 8270C/D; EPA 625	Hexachloroethane (HCE)
GC/MS	EPA 8270C/D; EPA 625	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D; EPA 625	Isophorone
GC/MS	EPA 8270C/D; EPA 625	Naphthalene
GC/MS	EPA 8270C/D; EPA 625	Nitrobenzene
GC/MS	EPA 8270C/D; EPA 625	N-Nitrosodimethylamine
GC/MS	EPA 8270C/D; EPA 625	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	EPA 8270C/D; EPA 625	N-nitrosodiphenylamine (NDPHA)
GC/MS	EPA 8270C/D; EPA 625	Pentachlorophenol
GC/MS	EPA 8270C/D; EPA 625	Phenanthrene
GC/MS	EPA 8270C/D; EPA 625	Phenol
GC/MS	EPA 8270C/D; EPA 625	Pyrene
GC/MS	EPA 8270C/D; EPA 625	Pyridine
GC/ECD	EPA 8081A/B	4,4'-DDD
GC/ECD	EPA 8081A/B	4,4'-DDE
GC/ECD	EPA 8081A/B	4,4'-DDT
GC/ECD	EPA 8081A/B	Aldrin
GC/ECD	EPA 8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	EPA 8081A/B	alpha-Chlordane
GC/ECD	EPA 8081A/B	beta-BHC (beta-HCH)
GC/ECD	EPA 8081A/B	delta-BHC (delta-HCH)

Non-Potable Water		
Technology	Method	Analyte
GC/ECD	EPA 8081A/B	Dieldrin
GC/ECD	EPA 8081A/B	Endosulfan I
GC/ECD	EPA 8081A/B	Endosulfan II
GC/ECD	EPA 8081A/B	Endosulfan sulfate
GC/ECD	EPA 8081A/B	Endrin
GC/ECD	EPA 8081A/B	Endrin aldehyde
GC/ECD	EPA 8081A/B	Endrin ketone
GC/ECD	EPA 8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	EPA 8081A/B	gamma-Chlordane
GC/ECD	EPA 8081A/B	Heptachlor
GC/ECD	EPA 8081A/B	Heptachlor epoxide
GC/ECD	EPA 8081A/B	Methoxychlor
GC/ECD	EPA 8081A/B	Chlordane (n.o.s.)
GC/ECD	EPA 8081A/B	Toxaphene
GC/ECD	EPA 8082A	Aroclor-1016
GC/ECD	EPA 8082A	Aroclor-1221
GC/ECD	EPA 8082A	Aroclor-1232
GC/ECD	EPA 8082A	Aroclor-1242
GC/ECD	EPA 8082A	Aroclor-1248
GC/ECD	EPA 8082A	Aroclor-1254
GC/ECD	EPA 8082A	Aroclor-1260
GC/ECD	EPA 8082A	Aroclor-1262
GC/ECD	EPA 8082A	Aroclor-1268
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4,5-TP (Silvex)
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichlorprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCP (Mecoprop)

Non-Potable Water		
Technology	Method	Analyte
HPLC/UV	EPA 8330A/B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A/B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A/B	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330A/B	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330A/B	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330A/B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Nitrotoluene (ONT)
HPLC/UV	EPA 8330A/B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/B	3-Nitrotoluene
HPLC/UV	EPA 8330A/B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A/B	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330A/B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330A/B	Nitrobenzene
HPLC/UV	EPA 8330A/B	Nitroglycerin
HPLC/UV	EPA 8330A/B	Nitroguanidine
HPLC/UV	EPA 8330A/B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330A/B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/B	PETN
GC/FID	FLPRO	Petroleum Range Organics
GC/FID	EPA 8015B/C	TPH DRO
GC/FID	EPA 8015B/C	TPH ORO
GC/FID	EPA 8015B/C	TPH GRO
GC/FID	RSK-175	Methane
GC/FID	RSK-175	Ethane
GC/FID	RSK-175	Ethene
GC/ECD	EPA 8011	1,2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1,2-Dibromo-3-chloropropane (DBCP)
HPLC/MS	EPA 6850	Perchlorate
ICP	EPA 6010B/C; EPA 200.7	Aluminum
ICP	EPA 6010B/C; EPA 200.7	Antimony
ICP	EPA 6010B/C; EPA 200.7	Arsenic
ICP	EPA 6010B/C; EPA 200.7	Barium

Non-Potable Water		
Technology	Method	Analyte
ICP	EPA 6010B/C; EPA 200.7	Beryllium
ICP	EPA 6010B/C; EPA 200.7	Boron
ICP	EPA 6010B/C; EPA 200.7	Cadmium
ICP	EPA 6010B/C; EPA 200.7	Calcium
ICP	EPA 6010B/C; EPA 200.7	Chromium, total
ICP	EPA 6010B/C; EPA 200.7	Cobalt
ICP	EPA 6010B/C; EPA 200.7	Copper
ICP	EPA 6010B/C; EPA 200.7	Iron
ICP	EPA 6010B/C; EPA 200.7	Lead
ICP	EPA 6010B/C; EPA 200.7	Magnesium
ICP	EPA 6010B/C; EPA 200.7	Manganese
CVAA	EPA 7470A; EPA 245.1	Mercury
ICP	EPA 6010B/C; EPA 200.7	Molybdenum
ICP	EPA 6010B/C; EPA 200.7	Nickel
ICP	EPA 6010B/C; EPA 200.7	Potassium
ICP	EPA 6010B/C; EPA 200.7	Selenium
ICP	EPA 6010B/C; EPA 200.7	Silver
ICP	EPA 6010B/C; EPA 200.7	Sodium
ICP	EPA 6010B/C; EPA 200.7	Strontium
ICP	EPA 6010B/C; EPA 200.7	Thallium
ICP	EPA 6010B/C; EPA 200.7	Tin
ICP	EPA 6010B/C; EPA 200.7	Titanium
ICP	EPA 6010B/C; EPA 200.7	Vanadium
ICP	EPA 6010B/C; EPA 200.7	Zinc
IC	EPA 300.0	Bromide
IC	EPA 300.0	Chloride
IC	EPA 300.0	Fluoride
IC	EPA 300.0	Nitrate
IC	EPA 300.0	Nitrite
IC	EPA 300.0	Sulfate
IC	EPA 9056A	Bromide
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride

Non-Potable Water		
Technology	Method	Analyte
IC	EPA 9056A	Nitrate
IC	EPA 9056A	Nitrite
IC	EPA 9056A	Sulfate
Colorimetric	SM 4500-NO ₂ B-2011	Nitrite as N
Titration	SM 2320 B-2011	Alkalinity
Colorimetric	SM 4500-NH ₃ G-2011	Ammonia
Probe	SM 5210 B-2011	BOD
Probe	SM 5210 B-2011	CBOD
Colorimetric	EPA 410.4	COD
UV/Vis	EPA 7196A SM 3500-Cr B-2011	Hexavalent Chromium
Colorimetric	EPA 353.2 MOD	Nitrocellulose
Colorimetric	EPA 353.2	Nitrate/Nitrite
Gravimetric	EPA 1664A	Oil and Grease
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	SM 4500-S ₂ F-2011	Sulfide
UV/Vis	SM 4500-P B5-2011	Total Phosphorus (as P)
UV/Vis	SM 4500-P E-2011	Ortho-Phosphate (as P)
TOC	EPA 9060A; SM 5310 C-2011	Total Organic Carbon
Gravimetric	SM 2540 C-2011	TDS
Gravimetric	SM 2540 D-2011	TSS
Colorimetric	EPA 9012A/B SM 4500-CN G-2011	Cyanide
Physical	EPA 1010A	Ignitability / Flashpoint
Physical	EPA 9095B	Paint Filter
Probe	EPA 9040B/C SM 4500-H ⁺ B-2011	pH(Corrosivity)
Preparation	Method	Type
Preparation	EPA 1311	TCLP
Preparation	EPA 3005A	Metals digestion
Preparation	EPA 3010A	Metals digestion
Preparation	EPA 3510C	Organics Liquid Extraction
Preparation	EPA 5030A/B	Purge and Trap Water

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	EPA 8260B	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	EPA 8260B	1,1,2-Trichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	EPA 8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	EPA 8260B	1,1-Dichloropropene
GC/MS	EPA 8260B	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B	1,2,3-Trichloropropane
GC/MS	EPA 8260B	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260B	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B	1,2-Dichlorobenzene
GC/MS	EPA 8260B	1,2-Dichloroethane (EDC)
GC/MS	EPA 8260B	1,2-Dichloropropane
GC/MS	EPA 8260B	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B	1,3-Dichlorobenzene
GC/MS	EPA 8260B	1,3-Dichloropropane
GC/MS	EPA 8260B	1,4-Dichlorobenzene
GC/MS	EPA 8260B	1,4-Dioxane
GC/MS	EPA 8260B	1-Chlorohexane
GC/MS	EPA 8260B	2,2-Dichloropropane
GC/MS	EPA 8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	EPA 8260B	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B	2-Chlorotoluene
GC/MS	EPA 8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	EPA 8260B	Acetone
GC/MS	EPA 8260B	Acetonitrile

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B	Acrolein
GC/MS	EPA 8260B	Acrylonitrile
GC/MS	EPA 8260B	Allyl chloride
GC/MS	EPA 8260B	Benzene
GC/MS	EPA 8260B	Bromobenzene
GC/MS	EPA 8260B	Bromochloromethane
GC/MS	EPA 8260B	Bromodichloromethane
GC/MS	EPA 8260B	Bromoform
GC/MS	EPA 8260B	Bromomethane
GC/MS	EPA 8260B	Carbon Disulfide
GC/MS	EPA 8260B	Carbon Tetrachloride
GC/MS	EPA 8260B	Chlorobenzene
GC/MS	EPA 8260B	Chloroethane
GC/MS	EPA 8260B	Chloroform
GC/MS	EPA 8260B	Chloromethane
GC/MS	EPA 8260B	Chloroprene
GC/MS	EPA 8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	EPA 8260B	cis-1,3-Dichloropropene
GC/MS	EPA 8260B	cis-1,4-Dichloro-2-butene
GC/MS	EPA 8260B	Cyclohexane
GC/MS	EPA 8260B	Dibromochloromethane
GC/MS	EPA 8260B	Dibromomethane
GC/MS	EPA 8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	EPA 8260B	Di-isopropyl ether
GC/MS	EPA 8260B	ETBE
GC/MS	EPA 8260B	Ethyl methacrylate
GC/MS	EPA 8260B	Ethylbenzene
GC/MS	EPA 8260B	Hexachlorobutadiene
GC/MS	EPA 8260B	Hexane
GC/MS	EPA 8260B	Iodomethane
GC/MS	EPA 8260B	Isobutyl alcohol
GC/MS	EPA 8260B	Isopropylbenzene (Cumene)

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B	m,p-Xylenes
GC/MS	EPA 8260B	Methacrylonitrile
GC/MS	EPA 8260B	Methyl Acetate
GC/MS	EPA 8260B	Methyl methacrylate
GC/MS	EPA 8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	EPA 8260B	Methylcyclohexane
GC/MS	EPA 8260B	Methylene Chloride, or Dichloromethane
GC/MS	EPA 8260B	Naphthalene
GC/MS	EPA 8260B	n-Butylbenzene
GC/MS	EPA 8260B	n-Propylbenzene
GC/MS	EPA 8260B	o-Xylene
GC/MS	EPA 8260B	p-Isopropyltoluene
GC/MS	EPA 8260B	Propionitrile
GC/MS	EPA 8260B	sec-Butylbenzene
GC/MS	EPA 8260B	Styrene
GC/MS	EPA 8260B	tert-Amyl methyl ether
GC/MS	EPA 8260B	tert-Butyl alcohol
GC/MS	EPA 8260B	tert-Butylbenzene
GC/MS	EPA 8260B	Tetrachloroethene (PCE; PERC)
GC/MS	EPA 8260B	Tetrahydrofuran
GC/MS	EPA 8260B	Toluene
GC/MS	EPA 8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	EPA 8260B	trans-1,3-Dichloropropene
GC/MS	EPA 8260B	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260B	Trichloroethene (TCE)
GC/MS	EPA 8260B	Trichlorofluoromethane (CFC-11)
GC/MS	EPA 8260B	Vinyl acetate
GC/MS	EPA 8260B	Vinyl Chloride (VC)
GC/MS	EPA 8260B	Xylenes (Total)
GC/MS	EPA 8270C/D	1,1'-Biphenyl
GC/MS	EPA 8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D	1,2,4-Trichlorobenzene

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/D	1,2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dioxane
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	EPA 8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	EPA 8270C/D	2,4-Dimethylphenol
GC/MS	EPA 8270C/D	2,4-Dinitrophenol
GC/MS	EPA 8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	EPA 8270C/D	2,6-Dichlorophenol
GC/MS	EPA 8270C/D	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 8270C/D	2-Nitrophenol (ONP)
GC/MS	EPA 8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	EPA 8270C/D	3-Methylphenol/4-Methylphenol
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 8270C/D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Methylphenol (p-Cresol)
GC/MS	EPA 8270C/D	4-Nitroaniline (PNA)
GC/MS	EPA 8270C/D	4-Nitrophenol (PNP)

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	Acenaphthene
GC/MS	EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetophenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 8270C/D	Benzidine
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270C/D	bis(2-Chloroethyl)ether (BCEE)
GC/MS	EPA 8270C/D	bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	EPA 8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/D	Dibenzofuran (DBF)
GC/MS	EPA 8270C/D	Diethyl phthalate (DEP)
GC/MS	EPA 8270C/D	Dimethyl phthalate (DMP)
GC/MS	EPA 8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	EPA 8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	EPA 8270C/D	Fluoranthene
GC/MS	EPA 8270C/D	Fluorene
GC/MS	EPA 8270C/D	Hexachlorobenzene (HCB)

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	Hexachlorobutadiene (HCBD)
GC/MS	EPA 8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	EPA 8270C/D	Hexachloroethane (HCE)
GC/MS	EPA 8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Naphthalene
GC/MS	EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	N-Nitrosodimethylamine
GC/MS	EPA 8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	EPA 8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenanthrene
GC/MS	EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/ECD	EPA 8081A/B	4,4'-DDD
GC/ECD	EPA 8081A/B	4,4'-DDE
GC/ECD	EPA 8081A/B	4,4'-DDT
GC/ECD	EPA 8081A/B	Aldrin
GC/ECD	EPA 8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	EPA 8081A/B	alpha-Chlordane
GC/ECD	EPA 8081A/B	beta-BHC (beta-HCH)
GC/ECD	EPA 8081A/B	delta-BHC (delta-HCH)
GC/ECD	EPA 8081A/B	Chlordane (n.o.s.)
GC/ECD	EPA 8081A/B	Dieldrin
GC/ECD	EPA 8081A/B	Endosulfan I
GC/ECD	EPA 8081A/B	Endosulfan II
GC/ECD	EPA 8081A/B	Endosulfan sulfate
GC/ECD	EPA 8081A/B	Endrin
GC/ECD	EPA 8081A/B	Endrin aldehyde
GC/ECD	EPA 8081A/B	Endrin ketone
GC/ECD	EPA 8081A/B	gamma-BHC (Lindane; gamma-HCH)

Solid and Chemical Materials		
Technology	Method	Analyte
GC/ECD	EPA 8081A/B	gamma-Chlordane
GC/ECD	EPA 8081A/B	Heptachlor
GC/ECD	EPA 8081A/B	Heptachlor epoxide
GC/ECD	EPA 8081A/B	Methoxychlor
GC/ECD	EPA 8081A/B	Toxaphene
GC/ECD	EPA 8082A	Aroclor-1016
GC/ECD	EPA 8082A	Aroclor-1221
GC/ECD	EPA 8082A	Aroclor-1232
GC/ECD	EPA 8082A	Aroclor-1242
GC/ECD	EPA 8082A	Aroclor-1248
GC/ECD	EPA 8082A	Aroclor-1254
GC/ECD	EPA 8082A	Aroclor-1260
GC/ECD	EPA 8082A	Aroclor-1262
GC/ECD	EPA 8082A	Aroclor-1268
HPLC/UV	EPA 8330A	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330A	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330A	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330A	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A	2-Nitrotoluene (ONT)
HPLC/UV	EPA 8330A	3-Nitrotoluene
HPLC/UV	EPA 8330A	3,5-Dinitroaniline
HPLC/UV	EPA 8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330A	Nitroglycerin
HPLC/UV	EPA 8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330A	Nitrobenzene
HPLC/UV	EPA 8330A	Nitroguanidine
HPLC/UV	EPA 8330A	PETN

Solid and Chemical Materials		
Technology	Method	Analyte
HPLC/UV	EPA 8330B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330B	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330B	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330B	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330B	2-Nitrotoluene (ONT)
HPLC/UV	EPA 8330B	3-Nitrotoluene
HPLC/UV	EPA 8330B	3,5-Dinitroaniline
HPLC/UV	EPA 8330B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330B	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330B	Nitroglycerin
HPLC/UV	EPA 8330B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330B	Nitrobenzene
HPLC/UV	EPA 8330B	Nitroguanidine
HPLC/UV	EPA 8330B	PETN
GC/FID	FLPRO	Petroleum Range Organics
GC/FID	EPA 8015B/C	TPH DRO
GC/FID	EPA 8015B/C	TPH ORO
GC/FID	EPA 8015B/C	TPH GRO
HPLC/MS	EPA 6850	Perchlorate
ICP	EPA 6010B/C	Aluminum
ICP	EPA 6010B/C	Antimony
ICP	EPA 6010B/C	Arsenic
ICP	EPA 6010B/C	Barium
ICP	EPA 6010B/C	Beryllium
ICP	EPA 6010B/C	Boron
ICP	EPA 6010B/C	Cadmium
ICP	EPA 6010B/C	Calcium
ICP	EPA 6010B/C	Chromium, total

Solid and Chemical Materials		
Technology	Method	Analyte
ICP	EPA 6010B/C	Cobalt
ICP	EPA 6010B/C	Copper
ICP	EPA 6010B/C	Iron
ICP	EPA 6010B/C	Lead
ICP	EPA 6010B/C	Magnesium
ICP	EPA 6010B/C	Manganese
CVAA	EPA 7471A/B	Mercury
ICP	EPA 6010B/C	Molybdenum
ICP	EPA 6010B/C	Nickel
ICP	EPA 6010B/C	Potassium
ICP	EPA 6010B/C	Selenium
ICP	EPA 6010B/C	Silver
ICP	EPA 6010B/C	Sodium
ICP	EPA 6010B/C	Strontium
ICP	EPA 6010B/C	Tin
ICP	EPA 6010B/C	Titanium
ICP	EPA 6010B/C	Thallium
ICP	EPA 6010B/C	Vanadium
ICP	EPA 6010B/C	Zinc
IC	EPA 9056A	Bromide
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate
IC	EPA 9056A	Nitrite
IC	EPA 9056A	Sulfate
UV/Vis	EPA 7196A	Hexavalent Chromium
TOC	Lloyd Kahn	Total Organic Carbon
Colorimetric	EPA 353.2 MOD	Nitrocellulose

Solid and Chemical Materials		
Technology	Method	Analyte
Colorimetric	EPA 9012A/B	Cyanide
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Physical	EPA 1010A	Ignitability/Flashpoint
Titration	EPA 9034	Sulfide
Probe	EPA 9045C/D	pH (Corrosivity)
Preparation	Method	Type
Preparation	EPA 1311	TCLP
Preparation	EPA 1312	SPLP
Preparation	NJ Modified 3060A	Hexavalent Chromium
Preparation	EPA 3050B	Metals Digestion
Preparation	EPA 3546	Organics Microwave Extraction
Preparation	EPA 3550B/C	Organics Sonication
Preparation	SM 2540 B-1997	Percent Solids (Percent Moisture)
Preparation	EPA 5035 /A	Purge and Trap Solid

Notes:

- 1) This laboratory offers commercial testing service.

Approved By:


 R. Douglas Leonard
 Chief Technical Officer

 Date: January 30, 2013

Re-issued: 1/30/13



State of Florida
Department of Health, Bureau of Public Health Laboratories
This is to certify that



E87646

EMPIRICAL LABORATORIES, LLC.
621 MAINSTREAM DRIVE SUITE 270
NASHVILLE, TN 37228

has complied with Florida Administrative Code 64E-1,
for the examination of environmental samples in the following categories

NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS,
NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS -
EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS - PESTICIDES-HERBICIDES-PCB'S, SOLID AND CHEMICAL MATERIALS - GENERAL
CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Public Health Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

Date Issued: July 01, 2013 Expiration Date: June 30, 2014



Victor Johnson, Director

Division of Emergency Preparedness and Community Support

DH Form 1697, 7/04

NON-TRANSFERABLE E87646-16-07/01/2013

Supersedes all previously issued certificates

**Laboratory Scope of Accreditation**

Attachment to Certificate #: E87646-16, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87646**EPA Lab Code: **TN00004****(615) 345-1115****E87646****Empirical Laboratories, LLC.****621 Mainstream Drive****Suite 270****Nashville, TN 37228**Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	4/6/2004
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	4/6/2004
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	EPA 8260	Volatile Organics	NELAP	6/16/2011
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	4/6/2004
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	4/6/2004
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	4/6/2004
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	10/5/2006
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	4/6/2004
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	6/16/2011
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8011	Volatile Organics	NELAP	8/15/2007
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8011	Volatile Organics	NELAP	8/15/2007
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	4/6/2004
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	4/6/2004
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	4/6/2004
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	6/16/2011
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	10/5/2006
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	4/6/2004
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	4/6/2004
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004

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Issue Date: 7/1/2013**Expiration Date: 6/30/2014**

Laboratory Scope of Accreditation

Page 2 of 20

Attachment to Certificate #: E87646-16, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87646**EPA Lab Code: **TN00004****(615) 345-1115****E87646****Empirical Laboratories, LLC.****621 Mainstream Drive****Suite 270****Nashville, TN 37228**Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	4/6/2004
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	4/6/2004
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	10/5/2006
1-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	6/16/2011
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	10/5/2006
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	4/6/2004
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	4/6/2004
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	6/16/2011
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	4/6/2004
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	4/6/2004
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	4/6/2004
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	4/6/2004
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	6/16/2011
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	10/5/2006
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	4/6/2004
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	6/16/2011
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	4/6/2004
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	4/6/2004
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	4/6/2004
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	6/16/2011
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	4/6/2004
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	4/6/2004

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Laboratory Scope of Accreditation

Page 3 of 20

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Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	4/6/2004
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	6/16/2011
2-Hexanone	EPA 8260	Volatile Organics	NELAP	4/6/2004
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	4/6/2004
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	4/6/2004
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	4/6/2004
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	4/6/2004
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	4/6/2004
3-Methylphenol (m-Cresol)	EPA 8270	Extractable Organics	NELAP	4/6/2004
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/6/2004
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	4/6/2004
4,4'-DDD	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4,4'-DDE	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4,4'-DDT	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	4/6/2004
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	4/6/2004
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	4/6/2004
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	4/6/2004
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	6/16/2011
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	4/6/2004
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/6/2004

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Laboratory Scope of Accreditation

Page 4 of 20

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Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	4/6/2004
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	4/6/2004
Acenaphthene	EPA 625	Extractable Organics	NELAP	4/6/2004
Acenaphthene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Acenaphthylene	EPA 625	Extractable Organics	NELAP	4/6/2004
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Acetone	EPA 8260	Volatile Organics	NELAP	4/6/2004
Acetonitrile	EPA 8260	Volatile Organics	NELAP	10/5/2006
Acetophenone	EPA 8270	Extractable Organics	NELAP	10/5/2006
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	4/6/2004
Aldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Alkalinity as CaCO ₃	SM 2320 B	General Chemistry	NELAP	3/4/2008
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aluminum	EPA 200.7	Metals	NELAP	4/6/2004
Aluminum	EPA 6010	Metals	NELAP	4/6/2004
Amenable cyanide	SM 4500-CN- G	General Chemistry	NELAP	3/4/2008
Ammonia as N	SM 4500-NH ₃ G	General Chemistry	NELAP	3/4/2008
Aniline	EPA 8270	Extractable Organics	NELAP	10/5/2006
Anthracene	EPA 625	Extractable Organics	NELAP	4/6/2004
Anthracene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Antimony	EPA 200.7	Metals	NELAP	4/6/2004
Antimony	EPA 6010	Metals	NELAP	4/6/2004
Aroclor-1016 (PCB-1016)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1221 (PCB-1221)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1232 (PCB-1232)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1242 (PCB-1242)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004

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Page 5 of 20

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Aroclor-1248 (PCB-1248)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1254 (PCB-1254)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1260 (PCB-1260)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Arsenic	EPA 200.7	Metals	NELAP	4/6/2004
Arsenic	EPA 6010	Metals	NELAP	4/6/2004
Atrazine	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	6/16/2011
Barium	EPA 200.7	Metals	NELAP	4/6/2004
Barium	EPA 6010	Metals	NELAP	4/6/2004
Benzaldehyde	EPA 8270	Extractable Organics	NELAP	6/16/2011
Benzene	EPA 624	Volatile Organics	NELAP	4/6/2004
Benzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Benidine	EPA 625	Extractable Organics	NELAP	4/6/2004
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	4/6/2004
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	4/6/2004
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	4/6/2004
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	4/6/2004
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	4/6/2004
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	10/5/2006
Beryllium	EPA 200.7	Metals	NELAP	4/6/2004
Beryllium	EPA 6010	Metals	NELAP	4/6/2004
beta-BHC (beta-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Biochemical oxygen demand	SM 5210 B	General Chemistry	NELAP	3/4/2008
Biphenyl	EPA 8270	Extractable Organics	NELAP	6/16/2011
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	4/6/2004
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	4/6/2004
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	4/6/2004

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Analyte	Method/Tech	Category	Certification Type	Effective Date
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	4/6/2004
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 625	Extractable Organics	NELAP	4/6/2004
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	4/6/2004
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	4/6/2004
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	4/6/2004
Boron	EPA 6010	Metals	NELAP	6/16/2011
Bromobenzene	EPA 8260	Volatile Organics	NELAP	6/16/2011
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	10/5/2006
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	4/6/2004
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Bromoform	EPA 624	Volatile Organics	NELAP	4/6/2004
Bromoform	EPA 8260	Volatile Organics	NELAP	4/6/2004
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	4/6/2004
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Cadmium	EPA 200.7	Metals	NELAP	4/6/2004
Cadmium	EPA 6010	Metals	NELAP	4/6/2004
Calcium	EPA 6010	Metals	NELAP	4/6/2004
Caprolactam	EPA 8270	Extractable Organics	NELAP	6/16/2011
Carbazole	EPA 8270	Extractable Organics	NELAP	4/6/2004
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	4/6/2004
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	4/6/2004
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	4/6/2004
Carbonaceous BOD (CBOD)	SM 5210 B	General Chemistry	NELAP	6/16/2011
Chemical oxygen demand	EPA 410.4	General Chemistry	NELAP	4/6/2004
Chlordane (tech.)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Chloride	EPA 300.0	General Chemistry	NELAP	4/6/2004
Chloride	EPA 9056	General Chemistry	NELAP	6/16/2011
Chlorobenzene	EPA 624	Volatile Organics	NELAP	4/6/2004
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Chloroethane	EPA 624	Volatile Organics	NELAP	4/6/2004
Chloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Chloroform	EPA 624	Volatile Organics	NELAP	4/6/2004
Chloroform	EPA 8260	Volatile Organics	NELAP	4/6/2004

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Page 7 of 20

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Chromium	EPA 200.7	Metals	NELAP	4/6/2004
Chromium	EPA 6010	Metals	NELAP	4/6/2004
Chromium VI	EPA 7196	General Chemistry	NELAP	4/6/2004
Chrysene	EPA 625	Extractable Organics	NELAP	4/6/2004
Chrysene	EPA 8270	Extractable Organics	NELAP	4/6/2004
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/6/2004
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	4/6/2004
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Cobalt	EPA 200.7	Metals	NELAP	4/6/2004
Cobalt	EPA 6010	Metals	NELAP	4/6/2004
Conductivity	EPA 120.1	General Chemistry	NELAP	4/6/2004
Copper	EPA 200.7	Metals	NELAP	4/6/2004
Copper	EPA 6010	Metals	NELAP	4/6/2004
Cyclohexane	EPA 8260	Volatile Organics	NELAP	6/16/2011
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
delta-BHC	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Dibenz(a,h)anthracene	EPA 625	Extractable Organics	NELAP	4/6/2004
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	4/6/2004
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	4/6/2004
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Dibromomethane	EPA 8260	Volatile Organics	NELAP	10/5/2006
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Dieldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	4/6/2004
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	4/6/2004
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	4/6/2004
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	4/6/2004
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004

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Issue Date: 7/1/2013**Expiration Date: 6/30/2014**

Laboratory Scope of Accreditation

Page 8 of 20

Attachment to Certificate #: E87646-16, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87646**EPA Lab Code: **TN00004****(615) 345-1115****E87646****Empirical Laboratories, LLC.****621 Mainstream Drive****Suite 270****Nashville, TN 37228**Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	4/6/2004
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endosulfan I	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endosulfan II	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endosulfan sulfate	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endrin aldehyde	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	10/5/2006
Ethylbenzene	EPA 624	Volatile Organics	NELAP	4/6/2004
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Fluoranthene	EPA 625	Extractable Organics	NELAP	4/6/2004
Fluoranthene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Fluorene	EPA 625	Extractable Organics	NELAP	4/6/2004
Fluorene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Fluoride	EPA 300.0	General Chemistry	NELAP	4/6/2004
Fluoride	EPA 9056	General Chemistry	NELAP	6/16/2011
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Gasoline range organics (GRO)	EPA 8015	Volatile Organics	NELAP	4/6/2004
Hardness (calc.)	EPA 200.7	General Chemistry	NELAP	4/6/2004
Heptachlor	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Heptachlor epoxide	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	4/6/2004
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004

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Issue Date: 7/1/2013**Expiration Date: 6/30/2014**

Laboratory Scope of Accreditation

Page 9 of 20

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State Laboratory ID: **E87646**EPA Lab Code: **TN00004****(615) 345-1115****E87646****Empirical Laboratories, LLC.****621 Mainstream Drive****Suite 270****Nashville, TN 37228**Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	4/6/2004
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	4/6/2004
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Hexachloroethane	EPA 625	Extractable Organics	NELAP	4/6/2004
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	4/6/2004
Ignitability	EPA 1010	General Chemistry	NELAP	4/6/2004
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	4/6/2004
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	10/5/2006
Iron	EPA 200.7	Metals	NELAP	4/6/2004
Iron	EPA 6010	Metals	NELAP	4/6/2004
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	10/5/2006
Isophorone	EPA 625	Extractable Organics	NELAP	4/6/2004
Isophorone	EPA 8270	Extractable Organics	NELAP	4/6/2004
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	10/5/2006
Kjeldahl nitrogen - total	EPA 351.2	General Chemistry	NELAP	4/6/2004
Lead	EPA 200.7	Metals	NELAP	4/6/2004
Lead	EPA 6010	Metals	NELAP	4/6/2004
Magnesium	EPA 200.7	Metals	NELAP	4/6/2004
Magnesium	EPA 6010	Metals	NELAP	4/6/2004
Manganese	EPA 200.7	Metals	NELAP	4/6/2004
Manganese	EPA 6010	Metals	NELAP	4/6/2004
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Mercury	EPA 245.1	Metals	NELAP	4/6/2004
Mercury	EPA 7470	Metals	NELAP	6/16/2011
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	10/5/2006
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Methyl acetate	EPA 8260	Volatile Organics	NELAP	6/16/2011
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	4/6/2004
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	4/6/2004
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	4/6/2004

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Issue Date: 7/1/2013**Expiration Date: 6/30/2014**

Laboratory Scope of Accreditation

Page 10 of 20

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State Laboratory ID: **E87646**EPA Lab Code: **TN00004****(615) 345-1115****E87646****Empirical Laboratories, LLC.****621 Mainstream Drive****Suite 270****Nashville, TN 37228**Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	10/5/2006
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Methylcyclohexane	EPA 8260	Volatile Organics	NELAP	6/16/2011
Methylene chloride	EPA 624	Volatile Organics	NELAP	4/6/2004
Methylene chloride	EPA 8260	Volatile Organics	NELAP	4/6/2004
Molybdenum	EPA 200.7	Metals	NELAP	4/6/2004
Molybdenum	EPA 6010	Metals	NELAP	4/6/2004
Naphthalene	EPA 625	Extractable Organics	NELAP	4/6/2004
Naphthalene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Naphthalene	EPA 8270	Extractable Organics	NELAP	4/6/2004
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	6/16/2011
Nickel	EPA 200.7	Metals	NELAP	4/6/2004
Nickel	EPA 6010	Metals	NELAP	4/6/2004
Nitrate	EPA 9056	General Chemistry	NELAP	6/16/2011
Nitrate as N	EPA 300.0	General Chemistry	NELAP	4/6/2004
Nitrite	EPA 300.0	General Chemistry	NELAP	6/16/2011
Nitrite	EPA 9056	General Chemistry	NELAP	6/16/2011
Nitrite as N	SM 4500-NO2-B	General Chemistry	NELAP	6/16/2011
Nitrobenzene	EPA 625	Extractable Organics	NELAP	4/6/2004
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	6/16/2011
Nitroglycerin	EPA 8330	Extractable Organics	NELAP	6/16/2011
Nitroglycerin	EPA 8332	Extractable Organics	NELAP	6/16/2011
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	4/6/2004
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	6/16/2011
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	4/6/2004
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	4/6/2004
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	4/6/2004
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	4/6/2004
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	6/16/2011
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	4/6/2004
Oil & Grease	EPA 1664A	General Chemistry	NELAP	4/6/2004
Orthophosphate as P	SM 4500-P E	General Chemistry	NELAP	3/4/2008
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	4/6/2004
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004

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Issue Date: 7/1/2013**Expiration Date: 6/30/2014**

Laboratory Scope of Accreditation

Page 11 of 20

Attachment to Certificate #: E87646-16, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87646**EPA Lab Code: **TN00004****(615) 345-1115****E87646****Empirical Laboratories, LLC.****621 Mainstream Drive****Suite 270****Nashville, TN 37228**Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Pentaerythritoltetranitrate (PETN)	EPA 8330	Extractable Organics	NELAP	6/16/2011
pH	EPA 9040	General Chemistry	NELAP	4/6/2004
pH	SM 4500-H+-B	General Chemistry	NELAP	3/4/2008
Phenanthrene	EPA 625	Extractable Organics	NELAP	4/6/2004
Phenanthrene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Phenol	EPA 625	Extractable Organics	NELAP	4/6/2004
Phenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
Phosphorus, total	SM 4500-P E	General Chemistry	NELAP	3/4/2008
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	6/16/2011
Potassium	EPA 6010	Metals	NELAP	4/6/2004
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	10/5/2006
Pyrene	EPA 625	Extractable Organics	NELAP	4/6/2004
Pyrene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Pyridine	EPA 8270	Extractable Organics	NELAP	4/6/2004
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	4/6/2004
Residue-filterable (TDS)	SM 2540 C	General Chemistry	NELAP	3/4/2008
Residue-nonfilterable (TSS)	SM 2540 D	General Chemistry	NELAP	3/4/2008
Residue-total	SM 2540 B	General Chemistry	NELAP	3/4/2008
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	6/16/2011
Selenium	EPA 200.7	Metals	NELAP	4/6/2004
Selenium	EPA 6010	Metals	NELAP	4/6/2004
Silver	EPA 200.7	Metals	NELAP	4/6/2004
Silver	EPA 6010	Metals	NELAP	4/6/2004
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Sodium	EPA 200.7	Metals	NELAP	4/6/2004
Sodium	EPA 6010	Metals	NELAP	4/6/2004
Styrene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Sulfate	EPA 300.0	General Chemistry	NELAP	4/6/2004
Sulfate	EPA 9056	General Chemistry	NELAP	6/16/2011
Sulfide	SM 4500-S F	General Chemistry	NELAP	7/1/2013
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	6/16/2011
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	4/6/2004
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	4/6/2004
Thallium	EPA 200.7	Metals	NELAP	4/6/2004

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Issue Date: 7/1/2013**Expiration Date: 6/30/2014**

Laboratory Scope of Accreditation

Page 12 of 20

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State Laboratory ID: **E87646**EPA Lab Code: **TN00004****(615) 345-1115****E87646****Empirical Laboratories, LLC.****621 Mainstream Drive****Suite 270****Nashville, TN 37228**Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Thallium	EPA 6010	Metals	NELAP	4/6/2004
Tin	EPA 200.7	Metals	NELAP	4/6/2004
Tin	EPA 6010	Metals	NELAP	4/6/2004
Titanium	EPA 6010	Metals	NELAP	6/16/2011
Toluene	EPA 624	Volatile Organics	NELAP	4/6/2004
Toluene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Total cyanide	EPA 9012	General Chemistry	NELAP	4/6/2004
Total nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	4/6/2004
Total organic carbon	EPA 9060	General Chemistry	NELAP	7/29/2004
Total organic carbon	SM 5310 C	General Chemistry	NELAP	3/4/2008
Total Petroleum Hydrocarbons (TPH)	FL-PRO	Extractable Organics	NELAP	4/6/2004
Total phenolics	EPA 420.4	General Chemistry	NELAP	7/1/2013
Toxaphene (Chlorinated camphene)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	4/6/2004
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/6/2004
trans-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	4/6/2004
trans-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	6/16/2011
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	10/5/2006
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	4/6/2004
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	4/6/2004
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Turbidity	EPA 180.1	General Chemistry	NELAP	4/6/2004
Vanadium	EPA 200.7	Metals	NELAP	4/6/2004
Vanadium	EPA 6010	Metals	NELAP	4/6/2004
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	10/5/2006
Vinyl chloride	EPA 624	Volatile Organics	NELAP	4/6/2004
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	4/6/2004
Xylene (total)	EPA 624	Volatile Organics	NELAP	4/6/2004
Xylene (total)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Zinc	EPA 200.7	Metals	NELAP	4/6/2004
Zinc	EPA 6010	Metals	NELAP	4/6/2004

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Laboratory Scope of Accreditation

Page 13 of 20

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State Laboratory ID: **E87646**EPA Lab Code: **TN00004****(615) 345-1115****E87646****Empirical Laboratories, LLC.****621 Mainstream Drive****Suite 270****Nashville, TN 37228**Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	EPA 8260	Volatile Organics	NELAP	6/16/2011
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	10/5/2006
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	6/16/2011
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	6/16/2011
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	10/5/2006
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	4/6/2004
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	4/6/2004
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	10/5/2006
1-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	6/16/2011
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	10/5/2006
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	4/6/2004
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004

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Issue Date: 7/1/2013**Expiration Date: 6/30/2014**

Laboratory Scope of Accreditation

Page 14 of 20

Attachment to Certificate #: E87646-16, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87646**EPA Lab Code: **TN00004****(615) 345-1115****E87646****Empirical Laboratories, LLC.****621 Mainstream Drive****Suite 270****Nashville, TN 37228**Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	4/6/2004
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	10/5/2006
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	4/6/2004
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	4/6/2004
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	4/6/2004
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	6/16/2011
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	6/16/2011
2-Hexanone	EPA 8260	Volatile Organics	NELAP	4/6/2004
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	4/6/2004
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	4/6/2004
3-Methylphenol (m-Cresol)	EPA 8270	Extractable Organics	NELAP	4/6/2004
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/6/2004
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	4/6/2004
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	4/6/2004
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	6/16/2011
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	4/6/2004
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	4/6/2004

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2013**Expiration Date: 6/30/2014**

Laboratory Scope of Accreditation

Page 15 of 20

Attachment to Certificate #: E87646-16, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87646**EPA Lab Code: **TN00004****(615) 345-1115****E87646****Empirical Laboratories, LLC.****621 Mainstream Drive****Suite 270****Nashville, TN 37228**Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	4/6/2004
Acenaphthene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Acetone	EPA 8260	Volatile Organics	NELAP	4/6/2004
Acetonitrile	EPA 8260	Volatile Organics	NELAP	10/5/2006
Acetophenone	EPA 8270	Extractable Organics	NELAP	10/5/2006
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	4/6/2004
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aluminum	EPA 6010	Metals	NELAP	4/6/2004
Aniline	EPA 8270	Extractable Organics	NELAP	10/5/2006
Anthracene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Antimony	EPA 6010	Metals	NELAP	4/6/2004
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Arsenic	EPA 6010	Metals	NELAP	4/6/2004
Atrazine	EPA 8270	Extractable Organics	NELAP	6/16/2011
Barium	EPA 6010	Metals	NELAP	4/6/2004
Benzaldehyde	EPA 8270	Extractable Organics	NELAP	6/16/2011
Benzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	10/5/2006

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Issue Date: 7/1/2013**Expiration Date: 6/30/2014**

Laboratory Scope of Accreditation

Page 16 of 20

Attachment to Certificate #: E87646-16, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87646**EPA Lab Code: **TN00004****(615) 345-1115****E87646****Empirical Laboratories, LLC.****621 Mainstream Drive****Suite 270****Nashville, TN 37228**Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Beryllium	EPA 6010	Metals	NELAP	4/6/2004
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Biphenyl	EPA 8270	Extractable Organics	NELAP	6/16/2011
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	4/6/2004
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	4/6/2004
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	4/6/2004
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	4/6/2004
Bromobenzene	EPA 8260	Volatile Organics	NELAP	6/16/2011
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	10/5/2006
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Bromoform	EPA 8260	Volatile Organics	NELAP	4/6/2004
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Cadmium	EPA 6010	Metals	NELAP	4/6/2004
Calcium	EPA 6010	Metals	NELAP	4/6/2004
Caprolactam	EPA 8270	Extractable Organics	NELAP	6/16/2011
Carbazole	EPA 8270	Extractable Organics	NELAP	4/6/2004
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	4/6/2004
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	4/6/2004
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Chloride	EPA 9056	General Chemistry	NELAP	4/6/2004
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Chloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Chloroform	EPA 8260	Volatile Organics	NELAP	4/6/2004
Chromium	EPA 6010	Metals	NELAP	4/6/2004
Chrysene	EPA 8270	Extractable Organics	NELAP	4/6/2004
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/6/2004
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Cobalt	EPA 6010	Metals	NELAP	4/6/2004
Copper	EPA 6010	Metals	NELAP	4/6/2004
Cyclohexane	EPA 8260	Volatile Organics	NELAP	6/16/2011
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	4/6/2004
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Dibromomethane	EPA 8260	Volatile Organics	NELAP	10/5/2006

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Issue Date: 7/1/2013**Expiration Date: 6/30/2014**

Laboratory Scope of Accreditation

Page 17 of 20

Attachment to Certificate #: E87646-16, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87646**EPA Lab Code: **TN00004****(615) 345-1115****E87646****Empirical Laboratories, LLC.****621 Mainstream Drive****Suite 270****Nashville, TN 37228**Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	4/6/2004
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	10/5/2006
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Fluoranthene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Fluorene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Fluoride	EPA 9056	General Chemistry	NELAP	4/6/2004
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	4/6/2004
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Hexachlorobenzene	EPA 8270	Extractable Organics,Pesticides-Herbicides-PCB' s	NELAP	4/6/2004
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics,Pesticides-Herbicides-PCB' s	NELAP	4/6/2004
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	4/6/2004
Ignitability	EPA 1010	General Chemistry	NELAP	4/6/2004
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	10/5/2006
Iron	EPA 6010	Metals	NELAP	4/6/2004
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	10/5/2006

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Issue Date: 7/1/2013**Expiration Date: 6/30/2014**

Laboratory Scope of Accreditation

Page 18 of 20

Attachment to Certificate #: E87646-16, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87646**EPA Lab Code: **TN00004****(615) 345-1115****E87646****Empirical Laboratories, LLC.****621 Mainstream Drive****Suite 270****Nashville, TN 37228**Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Isophorone	EPA 8270	Extractable Organics	NELAP	4/6/2004
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	10/5/2006
Lead	EPA 6010	Metals	NELAP	4/6/2004
Magnesium	EPA 6010	Metals	NELAP	4/6/2004
Manganese	EPA 6010	Metals	NELAP	4/6/2004
Mercury	EPA 7471	Metals	NELAP	4/6/2004
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	10/5/2006
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Methyl acetate	EPA 8260	Volatile Organics	NELAP	6/16/2011
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	10/5/2006
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Methylcyclohexane	EPA 8260	Volatile Organics	NELAP	6/16/2011
Methylene chloride	EPA 8260	Volatile Organics	NELAP	4/6/2004
Molybdenum	EPA 6010	Metals	NELAP	4/6/2004
Naphthalene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Naphthalene	EPA 8270	Extractable Organics	NELAP	4/6/2004
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	6/16/2011
Nickel	EPA 6010	Metals	NELAP	4/6/2004
Nitrate	EPA 9056	General Chemistry	NELAP	4/6/2004
Nitrite	EPA 9056	General Chemistry	NELAP	6/16/2011
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	4/6/2004
Nitroglycerin	EPA 8330	Extractable Organics	NELAP	6/16/2011
Nitroglycerin	EPA 8332	Extractable Organics	NELAP	6/16/2011
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	6/16/2011
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	4/6/2004
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	4/6/2004
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	6/16/2011
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	4/6/2004
Oil & Grease	EPA 1664A	General Chemistry	NELAP	6/16/2011
Paint Filter Liquids Test	EPA 9095	General Chemistry	NELAP	4/6/2004
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
Pentaerythritoltetranitrate (PETN)	EPA 8330	Extractable Organics	NELAP	6/16/2011

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Issue Date: 7/1/2013**Expiration Date: 6/30/2014**

Laboratory Scope of Accreditation

Page 19 of 20

Attachment to Certificate #: E87646-16, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87646**EPA Lab Code: **TN00004****(615) 345-1115****E87646****Empirical Laboratories, LLC.****621 Mainstream Drive****Suite 270****Nashville, TN 37228**Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Perchlorate	EPA 6850	General Chemistry	NELAP	7/1/2009
pH	EPA 9040	General Chemistry	NELAP	4/6/2004
pH	EPA 9045	General Chemistry	NELAP	4/6/2004
Phenanthrene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Phenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	6/16/2011
Potassium	EPA 6010	Metals	NELAP	4/6/2004
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	10/5/2006
Pyrene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Pyridine	EPA 8270	Extractable Organics	NELAP	4/6/2004
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	4/6/2004
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	6/16/2011
Selenium	EPA 6010	Metals	NELAP	4/6/2004
Silver	EPA 6010	Metals	NELAP	4/6/2004
Sodium	EPA 6010	Metals	NELAP	4/6/2004
Styrene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Sulfate	EPA 9056	General Chemistry	NELAP	4/6/2004
Synthetic Precipitation Leaching Procedure	EPA 1312	General Chemistry	NELAP	4/6/2004
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	6/16/2011
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	4/6/2004
Thallium	EPA 6010	Metals	NELAP	4/6/2004
Tin	EPA 6010	Metals	NELAP	4/6/2004
Toluene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Total cyanide	EPA 9012	General Chemistry	NELAP	4/6/2004
Total organic carbon	EPA 9060	General Chemistry	NELAP	7/29/2004
Total Petroleum Hydrocarbons (TPH)	FL-PRO	Extractable Organics	NELAP	4/6/2004
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	4/6/2004
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/6/2004
trans-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	4/6/2004
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	10/5/2006
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Vanadium	EPA 6010	Metals	NELAP	4/6/2004

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Issue Date: 7/1/2013**Expiration Date: 6/30/2014**



Laboratory Scope of Accreditation

Attachment to Certificate #: E87646-16, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87646**

EPA Lab Code: **TN00004**

(615) 345-1115

E87646

Empirical Laboratories, LLC.

621 Mainstream Drive

Suite 270

Nashville, TN 37228

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	10/5/2006
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	4/6/2004
Xylene (total)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Zinc	EPA 6010	Metals	NELAP	4/6/2004



State of Florida

Department of Health, Bureau of Public Health Laboratories
This is to certify that



E87832

MICROSEEPS, INC.
220 WILLIAM PITT WAY
PITTSBURGH, PA 15238

has complied with Florida Administrative Code 64E-1,
for the examination of environmental samples in the following categories

NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - VOLATILE ORGANICS

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Public Health Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

Date Issued: July 01, 2013 Expiration Date: June 30, 2014



Victor Johnson, Director

Division of Emergency Preparedness and Community Support

DH Form 1697, 7/04

NON-TRANSFERABLE E87832-15-07/01/2013

Supersedes all previously issued certificates



Laboratory Scope of Accreditation

Attachment to Certificate #: E87832-15, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87832

EPA Lab Code:

PA00076

(412) 826-5245

E87832

**Microseeps, Inc.
220 William Pitt Way
Pittsburgh, PA 15238**

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	4/1/2006
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/1/2006
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	4/1/2006
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	4/1/2006
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	4/1/2006
Acetone	EPA 8260	Volatile Organics	NELAP	4/1/2006
Alkalinity as CaCO ₃	SM 2320 B	General Chemistry	NELAP	4/1/2006
Benzene	EPA 8260	Volatile Organics	NELAP	4/1/2006
Bromobenzene	EPA 8260	Volatile Organics	NELAP	4/1/2006
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	4/1/2006
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	4/1/2006
Bromoform	EPA 8260	Volatile Organics	NELAP	4/1/2006
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	4/1/2006
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	4/1/2006
Chloride	EPA 9056	General Chemistry	NELAP	4/1/2006
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	4/1/2006
Chloroethane	EPA 8260	Volatile Organics	NELAP	4/1/2006
Chloroform	EPA 8260	Volatile Organics	NELAP	4/1/2006
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/1/2006

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2013

Expiration Date: 6/30/2014



Laboratory Scope of Accreditation

Attachment to Certificate #: E87832-15, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87832

EPA Lab Code:

PA00076

(412) 826-5245

E87832

**Microseeps, Inc.
220 William Pitt Way
Pittsburgh, PA 15238**

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	4/1/2006
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	4/1/2006
Dibromomethane	EPA 8260	Volatile Organics	NELAP	4/1/2006
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	4/1/2006
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	4/1/2006
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	4/1/2006
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	4/1/2006
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	4/1/2006
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	4/1/2006
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	4/1/2006
Methylene chloride	EPA 8260	Volatile Organics	NELAP	4/1/2006
Naphthalene	EPA 8260	Volatile Organics	NELAP	4/1/2006
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	4/1/2006
Nitrate	EPA 9056	General Chemistry	NELAP	4/1/2006
Nitrite	EPA 9056	General Chemistry	NELAP	4/1/2006
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	4/1/2006
pH	EPA 9040	General Chemistry	NELAP	5/5/2009
pH	SM 4500-H+-B	General Chemistry	NELAP	5/5/2009
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	4/1/2006
Styrene	EPA 8260	Volatile Organics	NELAP	4/1/2006
Sulfate	EPA 9056	General Chemistry	NELAP	4/1/2006
Sulfide	SM 4500-S F (19th/20th/21st Ed.)/TITR	General Chemistry	NELAP	5/5/2009
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	4/1/2006
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	4/1/2006
Toluene	EPA 8260	Volatile Organics	NELAP	4/1/2006
Total organic carbon	EPA 9060	General Chemistry	NELAP	4/1/2006
Total organic carbon	SM 5310 C	General Chemistry	NELAP	5/5/2009
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/1/2006
trans-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	4/1/2006
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	4/1/2006
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	4/1/2006
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	4/1/2006
Xylene (total)	EPA 8260	Volatile Organics	NELAP	4/1/2006

Appendix D
Laboratory Standard Operating Procedures

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

ORGANICS: SOP 202

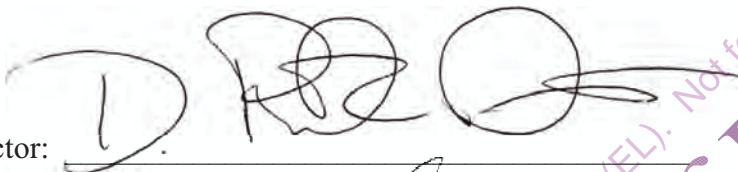
REVISION #: 26

EFFECTIVE DATE: 20130108

**GC/MS VOLATILES BY EPA METHOD E624 & SW846 METHOD 8260B
INCLUDING APPENDIX IX COMPOUNDS**

APPROVALS:

Lab Director:



Date: 01/08/2013

Data Quality Manager:



Date: 01/08/2013

Section Supervisor:



Date: 01/08/2013

Changes Summary

Revision 26, 01/08/13

- Addition to sections 10.6.1 and 13.4: **Note: While this concentration is a recommendation for SW-846 8260B (lesser amount could be used), it is required for USEPA 624. Any deviation from this concentration must be narrated in the reported data package.**

Revision 25, 09/26/12

- All references to Target have been updated to reflect Chemstation for data processing.
- Library reference has been updated to NIST98.L.
- Data review checklist has been updated to include a place for the sample number used to recalculate the concentration from raw area counts all the way to the final LIMS concentration.
- References to LCS have been updated to reflect BS.

Revision 24, 09/13/11

- This SOP is an update from Revision 23 dated 09/09/10.
- Section 9 has been updated with column and concentrator information.
- Section 10 has been updated with current standards mixtures.
- Section 13.1 has been updated to reflect calibration curve for analytes and surrogates.
- References to QSM 4.1 have been updated to QSM 4.2.

Revision 23, 09/09/10

- This SOP is an update from Revision 22 dated 09/30/09.
- Tables 1 and 2 have been updated with appropriate reference updates.
- Tables 5-7 have been added.

Revision 22, 9/30/09

- The SOP is an update from Revision 21 dated 09/11/08
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.2, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
3. Detection Limit
4. Scope of Application, Including components to be Analyzed
5. Summary of the Test Method
6. Definitions
7. Interferences
8. Safety
9. Equipment & Supplies
10. Reagents and Standards
11. Sample Collection, Preservation, Shipment, and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Data Analysis and Calculations
16. Method Performance
17. Pollution Prevention
18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Contingencies for Handling out-of-control or unacceptable data
20. Waste Management
21. References
22. Tables, Diagrams, Flowcharts and Validation Data

1. Identification of the Test Method

1.1 This SOP is compliant with methods – EPA Method 624 and SW-846 Method 8260B

0. Applicable Matrix or Matrices

2.1 This SOP is applicable to – The analysis of volatile organic compounds in a variety of matrices including but not limited to soils, sediments, ground and surface waters, aqueous sludge, oily wastes, etc.

0. Detection Limit: See **Table 1** of this SOP.

0. Scope of Application, Including components to be Analyzed

4.1 This SOP is based primarily on SW-846 Method 8260B. Methods SW-846 Method 8000B; *Federal Register* Method 624; and CLP Method for Volatiles have also been used in the development of this SOP. The analyses by these various methods are clearly defined in the respective regulatory manuals. A good understanding of these different methods is essential to the performance of each method. Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

0. Summary of the Test Method

5.1 After sample preparation, the sample is introduced into the GC/MS generally using purge and trap but sometimes using direct injection (see SW-846 Methods 5030B, 5035/5035A and 3585 for preparation). In purge and trap, the analytes are stripped from the sample using helium and trapped on an adsorbent tube. The tube is heated while being backflushed with helium to carry the analytes to the GC/MS system. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra of the sample. Analytes are quantitated relative to known standards using the internal standard method.

0. Definitions

6.1 Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

0. Interferences

7.1 Section 3.0 of SW-846 Method 8260B details interferences and potential problems which may be encountered when dealing with volatile analyses.

0. Safety

- 8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.

0. Equipment & Supplies

- 9.1 GC : HP 5890 or 6890, temperature programmable, suitable for split or splitless injection.
- 9.2 Column: HP-VOC, 30 meter x 0.2 mm I.D. 1.12 μ m film thickness silicon coated fused silica capillary column or equivalent.
- 9.3 M.S.: HP 5971, 5972 or 5973 capable of scanning 35 to 500 amu every one second or less, using 70 volts electron energy in electron impact ionization mode. The MS is capable of producing a mass spectrum for p-Bromofluorobenzene, BFB, which meets all tuning criteria for EPA methods [when 1 μ L (50 ng) of the GC/MS tuning standard is introduced to the GC.]
- 9.4 Purge and Trap Unit
- 9.4.1 Concentrators: Tekmar/Dohrmann 3000/3100 Sample Concentrator equipped with Supelco trap number 2-4920-U VOCARB 3000, or equivalent, providing good delivery for all target compounds.
- 0.3.1 Autosamplers: Varian Archon 51 position programmable autosampler with 5ml to 25ml water and heated soil capability.
- 9.5 Acquisition Software: HP Chemstation system interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.
- 9.6 Data Processing Software: HP Chemstation data system. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances of any EICP between specified time or scan-number limits. NIST98.L mass spectral library is installed.
- 9.7 Microsyringes—1.0, 5.0, 10, 25, 50, 100, 250, 500 and 1000 μ L.
- 9.8 Syringes—5, 25 and 50 mL, gas-tight with Luer end.
- 9.9 Balance - analytical, 0.0001 g; top-loading, 0.01 g.
- 9.10 Disposable pasteur pipets.
- 9.11 Volumetric flasks, Class A - 2 mL, 5 mL, 10 mL, 50 mL, 100 mL and 250 mL with ground-glass stoppers.
- 9.12 Wooden tongue depressors
- 9.13 Glass scintillation vials - 20mL with screw caps.
- 9.14 Latex or Nitrile Gloves
- 9.15 pH paper (measures pH from 0-14).

0. Reagents and Standards

- 10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:
- 10.2 Organic-free reagent water - obtained from the charcoal filter system in the VOA laboratory.
- 10.3 Methanol - Purge and trap grade (EM-Omnisolv EM-0482-6 or equivalent)
- 10.4 Methanol - suitable for use in gas chromatography (B&J Omnisolv MX0484- 1, or equivalent)
- 10.5 Sodium bisulfate, NaHSO_4 – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).
- 10.6 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label. The date they are opened is noted on the label and recorded in the LIMS system along with their lot number and vendor and given a sequential number. Each standard label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. Stock standards, when opened, have an expiration date of 6 months, **except for gas standards for South Carolina samples which have a one week expiration date**. All stocks and standards are stored in the freezer at a temperature of $-15^\circ\text{C} \pm 5^\circ\text{C}$ or less from the date they are received/prepared. The freezer temperature is monitored daily with a calibrated thermometer (annual calibration for liquid in glass and quarterly calibration for digital) and recorded with calibration correction in the VOA refrigerator/freezer logbook. Minimum and maximum temperatures are recorded after weekends/holidays. Makeup of common standards is detailed below. See standard information in LIMS system for makeup of other standards.
- 10.6.1 The Bromofluorobenzene (BFB) tuning standard is prepared as follows: Using a 50 μL syringe, 25 μL of standard (BFB @ 10000ng/ μL) is injected into a 5mL volumetric flask containing approximately 4.0mL P&T methanol (Vendor, Lot) and diluted to volume with same making a 50ng/ μL standard. After capping and inverting 3 times, the solution is transferred to a labeled teflon-lined, screw-capped vial and stored in the freezer at $-15^\circ\text{C} \pm 5^\circ\text{C}$ or less for up to a year (**1 week for South Carolina samples**). A direct injection of 1 μL (or equivalent purge) is used to tune the instrument. **Note: While this concentration is a recommendation for SW-846 8260B (lesser amount could be used), it is required for USEPA 624. Any deviation from this concentration must be narrated in the reported data package.**
- 10.6.2 The internal and surrogate standards are prepared as follows: Using the indicated syringe, the indicated amount of standard is injected into a 50 mL volumetric flask containing P&T methanol (Vendor, Lot) and diluted to volume with same making a 150ng/ μL standard. After capping and inverting 3 times, the solution is transferred to the Archon standard vial and stored under helium for 1 month or less. Each 8260/624 sample is automatically injected with 1 μL of this standard. (The internal standard/surrogate solution

may be replaced if the -50%-200% criteria exceeds in the CCV when calculated against the midpoint of the ICAL or previous CCV.)

Standard	Conc. (ng/μL)	Syringe (μL)	Amount (μL)
8260 ISTD Mix	2500	1000	3000
Surr. Mix	2500	1000	3000

10.6.3 Calibration standards are prepared from the vendor stock standards at appropriate concentrations as follows. Occasionally unusual compounds are added to the mix so it is best to check the LIMS for exact standard makeup. Note: for laboratory blank spikes (BS), alternate sources or lot numbers from the main calibration standard are used to make the BS standard.

10.6.3.1 Primary Standard: Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor, Lot) and diluted to volume with same to make a 100-500ng/μL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 100μg/L (5mL purge) standard is made using 50μL of this standard to 50mL of reagent water.

Stock Standard(CCV)	Conc (ng/μL)	Syringe(μL)	Amount(μL)	Final Conc (ng/μL)
Custom 2 Mix	2000-40000	250	200	200-4000
Ketones Mix	5000	250	160	400
Liquid mix	2000	250	200	200
Custom mix	2000-10000	250	200	200-1000
Gases (cat#30042)	2000	250	200	200
Oxygenates (CC2098.10)	2000-10000	250	200	200-1000

Additional compounds may be added such as Appendix IX. Refer to standard ID in LIMS system.

10.6.4 ICV/BS/Matrix Spike Mix: A second source standard is used to check the validity of the gas and primary calibration standards used in analyzing the calibration curve. Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor, Lot) and diluted to volume with same to make a 100-500ng/μL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 50μg/L ICV/BS/Matrix Spike is made using 25μL of this standard to 50mL of reagent water/Sample Matrix.

Stock Standard(ICV/BS)	Conc (ng/μL)	Syringe(μL)	Amount(μL)	Final Conc (ng/μL)
Custom 2 mix	2000-40000	100	100	100-2000
Oxygenates	2000-10000	100	100	100-500
Ketones	5000	100	80	200
Liquid mix	2000	100	100	100
Custom Mix	2000-10000	100	100	100-500

0. Sample Collection, Preservation, Shipment, and Storage

10.0 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

10.0 All water samples are stored in the “True” refrigerator in the VOA lab at a temperature of 4°C. All unpreserved soil samples in TerraCore or encores are stored in the freezer in the VOA lab. All soil samples in bulk jars or chemically preserved TerraCore are stored in the soil walk-in refrigerator at a temperature of 4°C. Non-preserved water volatile samples have a holding time of 7 days from date of sampling. Preserved water samples and soil volatile samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project). The temperature is monitored daily with a calibrated thermometer (annual calibration for liquid in glass and quarterly calibration for digital) and recorded with calibration correction in the VOA refrigerator/freezer logbook. The weekend temperature is monitored with a Min/Max thermometer and recorded upon arrival next business day.

0. Quality Control

12.1 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.

12.1 Internal Standards - All samples and QC are spiked with internals. See **Table 2** for acceptance criteria and corrective action.

12.2 Surrogates - All samples and QC are spiked with surrogates. See **Table 2** of this SOP for acceptance criteria and corrective action.

12.3 BS Sample - A BS is analyzed every 12 hour tune. To prepare the BS, a blank is spiked with standards prepared from an alternate vendor or lot number from the calibration standards. Note: the concentration of the BS will be 20 µg/L when analyzing 624 samples (QC Check Sample). See **Table 2** of this SOP for acceptance criteria and corrective action. **When analyzing samples for South Carolina the limits are 70-130% except for poor purgers which are 60-140%.**

12.4 Method Blanks - A method blank is analyzed every 12 hour tune. See **Table 2** of this SOP for acceptance criteria and corrective action..

12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for an MS/MSD with the BS standard. See **Table 2** of this SOP for acceptance criteria and corrective action. MS data evaluation must include the consideration of the following factors.

12.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. A water sample which was taken from the same VOA vial for the original sample and the MS/MSD should have very good RPDs unless there has been a method problem. Corrective action must be taken in the form of reanalysis if a method problem is indicated.

12.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was four or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.

12.5.3 MS vs. MSD - If a spiked compound has a problem in both the MS and MSD, review the BS and if acceptable no further action may be necessary since it is attributable to matrix effect.

12.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.

0. Calibration and Standardization

12.0 Quality Systems **SOP QS08** "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2 Chromatographic conditions – Refer to corresponding instrument maintenance log for current gas chromatograph, mass spectrometer, and concentrator conditions.

13.3 System Bakeout - Prior to analysis an instrument blank is analyzed.

NOTE: Further cleaning may be accomplished by backflushing the lines with methanol and then analyzing blanks overnight.

13.4 Tuning - Prior to any calibration or analysis, BFB tuning criteria must be met for a 1.0µL injection of the tuning standard. See **Table 5** of this SOP for acceptance criteria. Tune must be met every 12 hours sample analysis is to be performed (**every 24 hours for Federal Register Method 624 except for South Carolina which only allows 12 hours**). The mass spectrum of BFB is acquired as follows: by using the BFB method in Target (which uses three scans with background subtraction) to process the BFB data file. If the BFB tune does not pass criteria corrective action should be taken. **Note: While this concentration is a recommendation for SW-846 8260B (lesser amount could be used), it is required for USEPA 624. Any deviation from this concentration must be narrated in the reported data package.**

13.5 **Calibration:** Calibration standards are made up in water using the appropriate amount of the methanol standard. See the LIMS for preparation of standards. **Calibration for soils for South Carolina requires that 5mL of sodium bisulfate solution is added to each calibration standard made if the samples will be preserved with sodium bisulfate.** All manual calibration integrations must be approved by the section manager or designated peer reviewer.

13.5.1 Initial Calibration - An initial calibration curve at no less than five (six if using a quadratic curve fit) concentration levels for analytes and surrogates

must be analyzed and shown to meet the initial calibration criteria before any sample analysis may be performed. See **Table 2** of this SOP for acceptance criteria and corrective action. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All manual calibration integrations must be approved by the section manager or designated peer reviewer. Any response factors less than 0.050 must be supported by the mass spectrum of the lowest standard. **No quadratic curves for South Carolina.**

CCCs:	1,1-Dichloroethene	Toluene
	Chloroform	Ethylbenzene
	1,2-Dichloropropane	Vinyl chloride
SPCCs:	Chloromethane	0.10
	1,1-Dichloroethane	0.10
	Bromoform	0.10
	Chlorobenzene	0.30
	1,1,2,2-Tetrachloroethane	0.30

13.5.2 Initial Calibration Verification (ICV) - A second source standard is prepared at or near the CCV concentration and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. See **Table 2** of this SOP for acceptance criteria and corrective action. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All manual ICV integrations must be approved by the section manager or designated peer reviewer.

13.5.3 Continuing Calibration Verification (CCV) - A CCV is analyzed every 12 hour tune and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. See **Table 2** of this SOP for acceptance criteria and corrective action. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All manual CCV integrations must be approved by the section manager or designated peer reviewer.

NOTE: Acceptance criteria for method 624 consists of meeting recovery limits found in table 5 of the method for a QC check sample. This QC check sample is made from a separate source or lot number than the calibration standard at a concentration of 20 µg/L.

14. Procedure

14.1 BS - A BS is analyzed every 12 hour tune. Using standards prepared from an alternate vendor or lot number, blank water is spiked at the 50 µg/L (5mL/soil) or 10 µg/L (25mL) level. See **Table 2** of this SOP for acceptance criteria and corrective action.

Note: the concentration of the BS will be 20 µg/L when analyzing 624 samples (QC Check Sample).

14.2 Method Blank - Prior to sample analysis, the system must be shown to be free of contamination through analysis of a method blank. See **Table 2** of this SOP for acceptance criteria and corrective action.

14.3 Sample Analysis - Prior to analysis, the samples are prepared for chromatography using the appropriate sample preparation method (5mL water, 25mL water, low soil, high soil, etc.) See SOP 225 for preparation of a 5035 soil sample. For a 5mL/25mL water sample, use the following procedure:

14.3.1 Load the vial into the Archon autosampler in the expected position.

14.3.2 Program the Archon for the loaded vial range and necessary dilutions, making sure the programmed method is set for the same volume as the purge vessel on the front of the LSC 2000 or 3000/3100 and that the Chemstation sequence matches the Archon sequence. Note: TCLP samples are analyzed at a 10x dilution. One TCLP sample is spiked per batch at receipt of leachates.

14.3.3 After analysis of the sample has been completed, check the pH of the sample using pH paper and verify it to be less than a pH of 2 (recorded on the sequence log). If it is not, record the pH on the sequence log and generate a non-conformance report. The sample report will have to be qualified for preservation if the analysis is being performed more than 7 days after sampling. [Note: TCLP samples do not require a pH check.]

14.4 **Instrument sequence**

An example of a typical instrument sequence log follows:

- 1-BFB Tune (12:00 am)
- 2-CCV
- 3-BS
- 4-RL standard
- 5-Method Blank
- 6-Sample
- 7-Sample
- 8-Sample
- 9-Sample
- 10-Sample
- 11-Sample
- 12-Sample
- 13-Sample
- 14-Sample
- 15-Sample
- 16-Sample
- 17-Sample
- 18-Sample MS
- 19-Sample MSD
- 20-BFB (12:00pm - 12 hours since last BFB/CCV)
- 21-CCV
- 22-BS

23-Method Blank
24-Sample
25-Sample

14.5 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through the Chemstation data system. Quantitative measurements are performed using the calculations found in section 15.2 of this SOP. The following must be checked to determine if the sample will need any reanalysis or dilution. See **Table 2** of this SOP for acceptance criteria and corrective action. Formal data evaluation is detailed in SOP QS05. See **SOP QS07 for guidance on manual integrations.**

14.5.1 Internal Standards - Areas counts and retention times.

14.5.2 Surrogates – Recoveries and retention times.

Federal Register Method 624 contains no criteria for surrogate recovery.

Surrogate	WATER	SOIL
Dibromofluoromethane	85-120	80-125
1,2-Dichloroethane-d4	85-135	75-140
Toluene-d8	85-115	80-120
Bromofluorobenzene	80-120	80-125

14.5.3 Analyte concentration.

14.5.4 Qualitative identification based on spectrum and retention time.

15. Data Analysis and Calculations

15.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

15.2 Calculations:

13.1.1 The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.

A_{is} = Peak area (or height) of the internal standard.

C_s = Concentration of the analyte or surrogate.

C_{is} = Concentration of the internal standard.

15.2.2 Calibration verification involves the calculation of the percent drift (linear) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV RF} - \text{Average RF}) * 100}{\text{Average RF}}$$

where CCV RF is the response factor from the analysis of the verification standard and Average RF is the average response factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within $\pm 20\%$ for each CCC analyte, or for all target analytes if the CCCs are not target analytes, before any sample analyses may take place.

- 15.2.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to ug/L.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(V_s)(1000)}$$

where:

A_s = Area (or height) of the peak for the analyte in the sample.

A_{is} = Area (or height) of the peak for the internal standard.

C_{is} = Concentration of the internal standard in the volume purged in ug/L.

D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, $D = 1$. The dilution factor is always dimensionless.

V_i = For purge-and-trap analysis, V_i is not applicable and is set at 1.

\overline{RF} = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample purged (mL). If units of liters are used for this term, multiply the results by 1000.

- 15.2.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to ug/kg.]

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(W_s)(1000)}$$

where: A_s , A_{is} , C_{is} , D , and \overline{RF} are the same as for aqueous samples.

W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

16. Method Performance

- 16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-BS samples. The data is calculated for accuracy and precision requirements. The DOC form is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria.

17. Pollution Prevention

- 17.1 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

- 18.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. Contingencies for Handling out-of-control or unacceptable data

- 19.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. Table 2 within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20. Waste Management.

- 20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21. References

- 21.1 40 CFR, Part 136; Appendix A
- 21.2 Test Methods for Evaluating Solid Waste, SW-846, Third Edition and updates
- 21.3 National Environmental Laboratory Accreditation Conference; CH. 5, 2001
- 21.4 USACE, EM 200-1-3; Appendix 1; Shell, 2/2001
- 21.5 DOD Quality Systems Manual for Environmental Laboratories version 3, 3/2005
- 21.6 DOD Quality Systems Manual for Environmental Laboratories version 4.1, 4/2009
- 21.7 DOD Quality Systems Manual for Environmental Laboratories version 4.2, 10/2010

22. Tables, Diagrams, Flowcharts and Validation Data

- 22.1 Table 1, all parameters with DL(MDL)/LOD/LOQ(MRL).
- 22.2 Table 2, QA/QC summary table
- 22.3 Table 3, Technical Completeness / Accuracy Checklist

- 22.4 Table 4, Data Reviewers Checklist(s)
- 22.5 Table 5, BFB Tuning Criteria
- 22.6 Table 6, Analyst Checklist
- 22.7 Table 7, INTERNAL STANDARD ASSOCIATION

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Table 1 – DL/LOD/LOQ

Analyte	MDL/DL	LOD	MRL/LOQ	Units
1,1,1,2-Tetrachloroethane	1.25	2.50	5.00	ug/Kg
1,1,1-Trichloroethane (1,1,1-TCA)	1.25	2.50	5.00	ug/Kg
1,1,2,2-Tetrachloroethane	1.25	2.50	5.00	ug/Kg
1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)	2.50	5.00	10.0	ug/Kg
1,1,2-Trichloroethane	1.25	2.50	5.00	ug/Kg
1,1-Dichloroethane (1,1-DCA)	1.25	2.50	5.00	ug/Kg
1,1-Dichloroethene (1,1-DCE)	1.25	2.50	5.00	ug/Kg
1,1-Dichloropropene	1.25	2.50	5.00	ug/Kg
1,2,3-Trichlorobenzene	1.25	2.50	5.00	ug/Kg
1,2,3-Trichloropropane	1.25	2.50	5.00	ug/Kg
1,2,4-Trichlorobenzene	1.25	2.50	5.00	ug/Kg
1,2,4-Trimethylbenzene	1.25	2.50	5.00	ug/Kg
1,2-Dibromo-3-chloropropane (DBCP)	2.50	5.00	10.0	ug/Kg
1,2-Dibromoethane (EDB)	1.25	2.50	5.00	ug/Kg
1,2-Dichlorobenzene	1.25	2.50	5.00	ug/Kg
1,2-Dichloroethane (EDC)	1.25	2.50	5.00	ug/Kg
1,2-Dichloropropane	1.25	2.50	5.00	ug/Kg
1,3,5-Trimethylbenzene	1.25	2.50	5.00	ug/Kg
1,3-Dichlorobenzene	1.25	2.50	5.00	ug/Kg
1,3-Dichloropropane	1.25	2.50	5.00	ug/Kg
1,4-Dichlorobenzene	1.25	2.50	5.00	ug/Kg
2,2-Dichloropropane	1.25	2.50	5.00	ug/Kg
2-Butanone (Methyl ethyl ketone; MEK)	2.50	5.00	10.0	ug/Kg
2-Chlorotoluene	1.25	2.50	5.00	ug/Kg
2-Hexanone (Methyl butyl ketone; MBK)	1.25	2.50	5.00	ug/Kg
4-Chlorotoluene	1.25	2.50	5.00	ug/Kg
4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)	1.25	2.50	5.00	ug/Kg
Acetone	5.00	10.0	20.0	ug/Kg
Acrolein	5.00	10.0	20.0	ug/Kg
Acrylonitrile	5.00	10.0	20.0	ug/Kg
Benzene	1.25	2.50	5.00	ug/Kg
Bromobenzene	1.25	2.50	5.00	ug/Kg
Bromochloromethane	1.25	2.50	5.00	ug/Kg
Bromodichloromethane	1.25	2.50	5.00	ug/Kg
Bromoforn	1.25	2.50	5.00	ug/Kg
Bromomethane	2.50	5.00	10.0	ug/Kg
Carbon Disulfide	1.25	2.50	5.00	ug/Kg
Carbon Tetrachloride	1.25	2.50	5.00	ug/Kg
Chlorobenzene	1.25	2.50	5.00	ug/Kg
Chloroethane	2.50	5.00	10.0	ug/Kg
Chloroform	1.25	2.50	5.00	ug/Kg
Chloromethane	2.50	5.00	10.0	ug/Kg
cis-1,2-Dichloroethene (cis-1,2-DCE)	1.25	2.50	5.00	ug/Kg
cis-1,3-Dichloropropene	1.25	2.50	5.00	ug/Kg
Cyclohexane	1.25	2.50	5.00	ug/Kg
Dibromochloromethane	1.25	2.50	5.00	ug/Kg
Dibromomethane	1.25	2.50	5.00	ug/Kg
Dichlorodifluoromethane (CFC-12)	2.50	5.00	10.0	ug/Kg
Ethyl methacrylate	1.25	2.50	5.00	ug/Kg
Ethylbenzene	1.25	2.50	5.00	ug/Kg
Hexachlorobutadiene	1.25	2.50	5.00	ug/Kg
Iodomethane	5.00	10.0	20.0	ug/Kg
Isopropylbenzene (Cumene)	1.25	2.50	5.00	ug/Kg
Methyl Acetate	2.50	5.00	10.0	ug/Kg
Methyl methacrylate	1.25	2.50	5.00	ug/Kg
Methyl Tertiary Butyl Ether (MTBE)	1.25	2.50	5.00	ug/Kg
Methylcyclohexane	1.25	2.50	5.00	ug/Kg
Methylene Chloride, or Dichloromethane	2.50	5.00	10.0	ug/Kg

Analyte	MDL/DL	LOD	MRL/LOQ	Units
Naphthalene	1.25	2.50	5.00	ug/Kg
n-Butylbenzene	1.25	2.50	5.00	ug/Kg
n-Propylbenzene	1.25	2.50	5.00	ug/Kg
p-Isopropyltoluene	1.25	2.50	5.00	ug/Kg
sec-Butylbenzene	1.25	2.50	5.00	ug/Kg
Styrene	1.25	2.50	5.00	ug/Kg
tert-Butylbenzene	1.25	2.50	5.00	ug/Kg
Tetrachloroethene (PCE; PERC)	1.25	2.50	5.00	ug/Kg
Toluene	1.25	2.50	5.00	ug/Kg
trans-1,2-Dichloroethene (trans-1,2-DCE)	1.25	2.50	5.00	ug/Kg
trans-1,3-Dichloropropene	1.25	2.50	5.00	ug/Kg
Trichloroethene (TCE)	1.25	2.50	5.00	ug/Kg
Trichlorofluoromethane (CFC-11)	2.50	5.00	10.0	ug/Kg
Vinyl acetate	2.50	5.00	10.0	ug/Kg
Vinyl Chloride (VC)	2.50	5.00	10.0	ug/Kg
m,p-Xylene	2.50	5.00	10.0	ug/Kg
o-Xylene	1.25	2.50	5.00	ug/Kg
1,1,1,2-Tetrachloroethane	0.25	0.50	1.00	ug/L
1,1,1-Trichloroethane (1,1,1-TCA)	0.25	0.50	1.00	ug/L
1,1,2,2-Tetrachloroethane	0.25	0.50	1.00	ug/L
1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)	0.50	1.00	2.00	ug/L
1,1,2-Trichloroethane	0.25	0.50	1.00	ug/L
1,1-Dichloroethane (1,1-DCA)	0.25	0.50	1.00	ug/L
1,1-Dichloroethene (1,1-DCE)	0.25	0.50	1.00	ug/L
1,1-Dichloropropene	0.25	0.50	1.00	ug/L
1,2,3-Trichlorobenzene	0.25	0.50	1.00	ug/L
1,2,3-Trichloropropane	0.50	1.00	2.00	ug/L
1,2,4-Trichlorobenzene	0.25	0.50	1.00	ug/L
1,2,4-Trimethylbenzene	0.25	0.50	1.00	ug/L
1,2-Dibromo-3-chloropropane (DBCP)	0.50	1.00	2.00	ug/L
1,2-Dibromoethane (EDB)	0.25	0.50	1.00	ug/L
1,2-Dichlorobenzene	0.25	0.50	1.00	ug/L
1,2-Dichloroethane (EDC)	0.25	0.50	1.00	ug/L
1,2-Dichloropropane	0.25	0.50	1.00	ug/L
1,3,5-Trimethylbenzene	0.25	0.50	1.00	ug/L
1,3-Dichlorobenzene	0.25	0.50	1.00	ug/L
1,3-Dichloropropane	0.25	0.50	1.00	ug/L
1,4-Dichlorobenzene	0.25	0.50	1.00	ug/L
1-Chlorohexane	0.50	1.00	2.00	ug/L
2,2-Dichloropropane	0.25	0.50	1.00	ug/L
2-Butanone (Methyl ethyl ketone; MEK)	2.50	5.00	10.0	ug/L
2-Chloroethyl vinyl ether	1.25	2.50	5.00	ug/L
2-Chlorotoluene	0.25	0.50	1.00	ug/L
2-Hexanone (Methyl butyl ketone; MBK)	1.25	2.50	5.00	ug/L
4-Chlorotoluene	0.25	0.50	1.00	ug/L
4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)	1.25	2.50	5.00	ug/L
Acetone	2.50	5.00	10.0	ug/L
Acrolein	1.25	2.50	5.00	ug/L
Acrylonitrile	2.50	5.00	10.0	ug/L
Benzene	0.25	0.50	1.00	ug/L
Bromobenzene	0.25	0.50	1.00	ug/L
Bromochloromethane	0.25	0.50	1.00	ug/L
Bromodichloromethane	0.25	0.50	1.00	ug/L
Bromoform	0.25	0.50	1.00	ug/L
Bromomethane	0.50	1.00	2.00	ug/L
Carbon Disulfide	0.25	0.50	1.00	ug/L
Carbon Tetrachloride	0.25	0.50	1.00	ug/L
Chlorobenzene	0.25	0.50	1.00	ug/L
Chloroethane	0.50	1.00	2.00	ug/L
Chloroform	0.25	0.50	1.00	ug/L
Chloromethane	0.25	0.50	1.00	ug/L
cis-1,2-Dichloroethene (cis-1,2-DCE)	0.25	0.50	1.00	ug/L

Analyte	MDL/DL	LOD	MRL/LOQ	Units
cis-1,3-Dichloropropene	0.25	0.50	1.00	ug/L
Cyclohexane	0.25	0.50	1.00	ug/L
Dibromochloromethane	0.25	0.50	1.00	ug/L
Dibromomethane	0.25	0.50	1.00	ug/L
Dichlorodifluoromethane (CFC-12)	0.50	1.00	2.00	ug/L
Di-isopropyl ether	0.25	0.50	1.00	ug/L
ETBE	0.25	0.50	1.00	ug/L
Ethyl methacrylate	0.25	0.50	1.00	ug/L
Ethylbenzene	0.25	0.50	1.00	ug/L
Hexachlorobutadiene	0.25	0.50	1.00	ug/L
Iodomethane	0.25	0.50	1.00	ug/L
Isopropylbenzene (Cumene)	0.25	0.50	1.00	ug/L
Methyl Acetate	0.50	1.00	2.00	ug/L
Methyl methacrylate	0.25	0.50	1.00	ug/L
Methyl Tertiary Butyl Ether (MTBE)	0.25	0.50	1.00	ug/L
Methylcyclohexane	0.25	0.50	1.00	ug/L
Methylene Chloride, or Dichloromethane	0.50	1.00	2.00	ug/L
Naphthalene	0.25	0.50	1.00	ug/L
n-Butylbenzene	0.25	0.50	1.00	ug/L
n-Propylbenzene	0.25	0.50	1.00	ug/L
p-Isopropyltoluene	0.25	0.50	1.00	ug/L
sec-Butylbenzene	0.25	0.50	1.00	ug/L
Styrene	0.25	0.50	1.00	ug/L
t-Butyl alcohol	1.25	2.50	5.00	ug/L
tert-Amyl methyl ether	2.50	5.00	10.0	ug/L
tert-Butylbenzene	0.25	0.50	1.00	ug/L
Tetrachloroethene (PCE; PERC)	0.25	0.50	1.00	ug/L
Tetrahydrofuran	1.25	2.50	5.00	ug/L
Toluene	0.25	0.50	1.00	ug/L
trans-1,2-Dichloroethene (trans-1,2-DCE)	0.25	0.50	1.00	ug/L
trans-1,3-Dichloropropene	0.25	0.50	1.00	ug/L
Trichloroethene (TCE)	0.25	0.50	1.00	ug/L
Trichlorofluoromethane (CFC-11)	0.50	1.00	2.00	ug/L
Vinyl acetate	1.25	2.50	5.00	ug/L
Vinyl Chloride (VC)	0.50	1.00	2.00	ug/L
m,p-Xylene	0.50	1.00	2.00	ug/L
o-Xylene	0.25	0.50	1.00	ug/L

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Method 8260B)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f of DoD QSM 4.2).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL determination	Initial method demonstration required for some states – not required for DoD	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis then quarterly verification.	See Box D-13 of DoD QSM 4.2			
LOQ establishment and verification	Prior to initial analysis then quarterly verification.	See Box D-14 of DoD QSM 4.2			
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to table 5 of this SOP.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	<p>1. Average response factor (RF) for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane.</p> <p>2. RSD for RFs for CCCs: VOCs $\leq 30\%$ and one option below: Option 1: RSD for each analyte $\leq 15\%$; Option 2: linear least squares regression $r \geq 0.995$; Option 3: non-linear regression–coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order).</p>	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin for DoD projects.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260B) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within $\pm 20\%$ of true value. [$\pm 25\%$ for non-DoD 8260B;]	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the sequence CCV is used.	NA.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units. Note - retention times may be updated based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping).	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ± 0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. %Difference/Drift for all target compounds and surrogates: VOCs $\leq 20\%D$ (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration). [$\pm 20\%$ for CCCs only non-DoD 8260B]	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since last acceptable CCV. [For non-DoD 8260B, if CCCs exceed, evaluate all analytes for 20%D and qualify as above]	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed, holding time has been exceeded or client has approved reporting.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260B) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal standards verification	Every field sample, standard, and QC sample.	Retention time \pm 30 seconds from retention time of the midpoint standard in the ICAL or daily CCV; EICP area within -50% to +100% of ICAL midpoint standard or daily CCV.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply qualifier to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method blank	One per preparatory batch.	No analytes detected $> \frac{1}{2}$ RL and $> \frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected $> \text{RL/LOQ}$	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
BS containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by client or DoD (appendix G), if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean BS recovery.	Correct problem, then reprep and reanalyze the BS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid BS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix (see Box D-7).	Use BS criteria, above.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the BS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation, use BS acceptance criteria above. MSD or sample duplicate: $\text{RPD} \leq 30\%$ or client specified limit (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260B) (continued)

QC Check	Minimum Frequency	Acceptance Criteria			Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	Surrogate	WATER	SOIL	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply qualifier to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
		Dibromofluoromethane	85-120	80-125			
		1,2-Dichloroethane-d4	85-135	75-140			
		Toluene-d8	85-115	80-120			
		Bromofluorobenzene	80-120	80-125			
		QC acceptance criteria specified by DoD (above) or Client. Otherwise, in-house control limits may be used. No limits specified for Method 624.					
Results reported between DL and LOQ	NA.	NA.			NA.	Apply J-flag to all results between DL and LOQ.	

Table 3, Technical Completeness / Accuracy Checklist

- 13. Were all the QC check elements analyzed – refer to Table 2 of the SOP
- 13. Were the QC criteria met
- 13. In cases of failures, was there an NCR written
- 13. Were all manual integrations signed
- 13. Were dilution factors applied correctly
- 13. Was there supervisory approval for manual integrations on standards and QC samples
- 13. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
- 13. If the data was entered into LIMS manually, was a check of all entered values performed
- 13. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
- 13. Were proper data qualifiers applied to the data in LIMS
- 13. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

- 0. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
- 0. Were QA objectives met and for failures were the appropriate actions taken
- 0. For direct uploads to LIMS, did a subset cross check match the raw data
- 0. Did all the manual entries into LIMS match the raw data
- 0. Were there appropriate signatures and documentation on the raw data
- 0. Were appropriate LIMS flags used
- 0. Were manual integrations signed
- 0. Were manual integrations for calibration and QC samples approved by supervisor
- 0. Were manual calculations verified

Table 5, Tuning Criteria

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

Table 6, ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s)/Target ID:	
Sequence Number/Calibration Number:	
Method: 8260B/624, 8270C/8270D/625	NCR#

QA/QC Item	Yes	No	NA	2 nd Check
0. Was the autosampler tray verified against the sequence file?	_____	_____	_____	_____
2. Is the BFB/DFTPP tune performed every 12 hours and is the tuning criteria met? For 8270 regular (not low PAH), have tailing and breakdown criteria been met?	_____	_____	_____	_____
3. Are the % RSDs within 20% or 0.995 linear corr or 0.990 quadratic COD for all analytes in the initial calibration? Are SPCC response factor criteria met? Is recal. of low stds within 50%-150% (preferred). Retention times checked for compounds with the same spectra (ex. Dichlorobenzenes). Concentrations checked for compounds with different conc. (ex. m/p-xylene, ketones,etc.).	_____	_____	_____	_____
4. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met (+/-20% DoD, +/-25% method)? Are SPCC response factor criteria met?	_____	_____	_____	_____
5. Does the Continuing Calibration Standard (CCV) meet the $\pm 20\%$ difference criteria and IS within 50%-200% of calibration curve midpoint?	_____	_____	_____	_____
6. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the RL (LOD for DoD except phthalates)?	_____	_____	_____	_____
6. Are the BS, BSD, MS, MSD within control limits and run at the desired frequency?	_____	_____	_____	_____
8. Are all sample holding times met, analytes within calibration range, IS areas within 50%-200% of CCV response and surrogate recoveries within limits?	_____	_____	_____	_____
9. Sample_____shows calculation verified from raw areas to final LIMS concentration.	_____	_____	_____	_____
0. Data uploaded to Element with correct analysts reflected?	_____	_____	_____	_____
	_____	_____	_____	_____
	_____	_____	_____	_____

Comments on any "No" response:

Data uploaded?

Data Qualified?

Primary-Level Review: _____ Date: _____

Second-Level Review: _____ Date: _____

Table 7, Internal Standard Association

Analyte	Internal Standard	Analyte	Internal Standard
1,1,1-Trichloroethane	Fluorobenzene	1,1,1,2-Tetrachloroethane	d5-Chlorobenzene
1,1,2-Trichloro-1,2,2-trifluoroethane	Fluorobenzene	1,1,2-Trichloroethane	d5-Chlorobenzene
1,1-Dichloroethane	Fluorobenzene	1,2,3-Trichloropropane	d5-Chlorobenzene
1,1-Dichloropropene	Fluorobenzene	1,2-Dibromochloroethane (EDB)	d5-Chlorobenzene
1,2-Dichloroethane	Fluorobenzene	1,3-Dichloropropane	d5-Chlorobenzene
1,2-Dichloroethane-d4	Fluorobenzene	1-Chlorohexane	d5-Chlorobenzene
1,2-Dichloroethane (total)	Fluorobenzene	2-Hexanone	d5-Chlorobenzene
1,2-Dichloropropane	Fluorobenzene	Bromofluorobenzene	d5-Chlorobenzene
1,4-Dioxane	Fluorobenzene	Bromoforn	d5-Chlorobenzene
2,2-Dichloropropane	Fluorobenzene	Chlorobenzene	d5-Chlorobenzene
2-Butanone	Fluorobenzene	Chlorobenzene-d5	d5-Chlorobenzene
2-Chloroethyl vinyl ether	Fluorobenzene	Dibromochloromethane	d5-Chlorobenzene
4-Methyl-2-pentanone	Fluorobenzene	Ethyl Methacrylate	d5-Chlorobenzene
Acetaldehyde	Fluorobenzene	Ethylbenzene	d5-Chlorobenzene
Acetone	Fluorobenzene	m,p-Xylene	d5-Chlorobenzene
Acetonitrile	Fluorobenzene	Methacrylonitrile	d5-Chlorobenzene
Acrolein	Fluorobenzene	o-Xylene	d5-Chlorobenzene
Acrylonitrile	Fluorobenzene	Styrene	d5-Chlorobenzene
Allyl chloride	Fluorobenzene	Tetrachloroethane	d5-Chlorobenzene
Benzene	Fluorobenzene	Toluene	d5-Chlorobenzene
Bromochloromethane	Fluorobenzene	Toluene-d8	d5-Chlorobenzene
Bromodichloromethane	Fluorobenzene	trans-1,3-Dichloropropene	d5-Chlorobenzene
Bromomethane	Fluorobenzene	Xylenes (total)	d5-Chlorobenzene
Carbon disulfide	Fluorobenzene	1,1,2,2-Tetrachloroethane	1,4-dichlorobenzene-d4
Carbon tetrachloride	Fluorobenzene	1,2,3-Trichlorobenzene	1,4-dichlorobenzene-d4
Chloroethane	Fluorobenzene	1,2,4-Trichlorobenzene	1,4-dichlorobenzene-d4
Chloroform	Fluorobenzene	1,2,4-Trimethylbenzene	1,4-dichlorobenzene-d4
Chloromethane	Fluorobenzene	1,2-Dibromo-3-chloropropane	1,4-dichlorobenzene-d4
Chloroprene	Fluorobenzene	1,2-Dichlorobenzene	1,4-dichlorobenzene-d4
cis-1,2-Dichloroethane	Fluorobenzene	1,3,5-Trimethylbenzene	1,4-dichlorobenzene-d4
cis-1,3-Dichloropropene	Fluorobenzene	1,3-Dichlorobenzene	1,4-dichlorobenzene-d4
Cyclohexane	Fluorobenzene	1,4-Dichlorobenzene	1,4-dichlorobenzene-d4
Dibromofluoromethane	Fluorobenzene	1,4-Dichlorobenzene-d4	1,4-dichlorobenzene-d4
Dibromomethane	Fluorobenzene	2-Chlorotoluene	1,4-dichlorobenzene-d4
Dichlorodifluoromethane	Fluorobenzene	4-Chlorotoluene	1,4-dichlorobenzene-d4
Diisopropyl Ether	Fluorobenzene	Bromobenzene	1,4-dichlorobenzene-d4
Ethyl tert-Butyl Ether	Fluorobenzene	cis-1,4-Dichloro-2-butene	1,4-dichlorobenzene-d4
Fluorobenzene	Fluorobenzene	Hexachlorobutadiene	1,4-dichlorobenzene-d4
Hexane	Fluorobenzene	Naphthalene	1,4-dichlorobenzene-d4
Iodomethane	Fluorobenzene	n-Butylbenzene	1,4-dichlorobenzene-d4
Isobutyl alcohol	Fluorobenzene	n-Propylbenzene	1,4-dichlorobenzene-d4
Isopropylbenzene	Fluorobenzene	p-Isopropyltoluene	1,4-dichlorobenzene-d4
Methyl Acetate	Fluorobenzene	sec-Butylbenzene	1,4-dichlorobenzene-d4
Methyl Methacrylate	Fluorobenzene	tert-Butylbenzene	1,4-dichlorobenzene-d4
Methyl t-Butyl Ether	Fluorobenzene	trans-1,4-Dichloro-2-butene	1,4-dichlorobenzene-d4
Methylcyclohexane	Fluorobenzene		
Methylene chloride	Fluorobenzene		
Propionitrile	Fluorobenzene		
t-Butyl alcohol	Fluorobenzene		
Tert-Amyl Methyl Ether	Fluorobenzene		
Tetrahydrofuran	Fluorobenzene		
trans-1,2-Dichloroethane	Fluorobenzene		
Trichloroethene	Fluorobenzene		
Trichlorofluoromethane	Fluorobenzene		
Vinyl acetate	Fluorobenzene		
Vinyl chloride	Fluorobenzene		

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

INORGANICS: SOP100

REVISION #: 25

EFFECTIVE DATE: 20131219

METALS DIGESTION/PREPARATION

References:

Methods 3005A, 3010A, 3050B and EPA 200.7

APPROVALS:

Lab Director: _____

Date: 12/19/2013

Data Quality Manager: _____

Date: 12/19/2013

Section Supervisor: _____

Date: 12/19/2013

Changes Summary

Revision 25, 12/19/2013

- Section VII.2.A.1.a: Removed residual reference to CLP preparation.
- Section VII.2.B.: Updated spike solution preparation information.
- Section IX. Updated information on digestion vessels and filter apparatus.
- Section IX.C.1. References to 200.7 removed.
- Section IX.C.2. Added 200.7 digestion process.
- Section IX.C.3: Previously IX.C.2 for Method 3010A.
- Attachment: Circle Sheet updated for spike SM-391-001 and separation of 3005A/200.7.

Revision 24, 09/03/2013

- Removed any references CLP and Standard Methods.
- Combined Method 3005A and EPA 200.7 digestion instructions.
- Attached Eppendorf/Solution Circle Sheets used for daily digestions.

Revision 23, 03/05/12

- **The SOP is an update from** Revision 22 11/17/10
- Removed any references to using watch glasses
- Added a note that the method does not require digestion for dissolved metals. Analysts digest dissolved samples only when selenium is requested.
- Changed references to Hot plate, Thermolyne Eimarec-3 to Hot block, Environmental Express
- Removed Reagents used for oil method that is no longer performed.
- Removed statement that analyst must initial and date each entry made in LIMS.
- Changed LCS to Blank Spike

Revision 22, 11/17/10

- The SOP is an update from Revision 21 dated 9/1/10
- Revised to add the need for matrix spike duplicates to be digested and analyzed for TCLP extracts.
- Requirement to hold samples 24 hours after in-house preservation was added to section III.

Revision 21, 9/1/10

- The SOP is an update from Revision 20 dated 04/27/10
- The SOP has been found to be up-to-date with Standard Methods 21st edition.
- Reference to adjusting filtrate volume for method 3030C has been removed.
- References to bound logbooks have been replaced with LIMS references.

Revision 20, 4/27/10

- The SOP is an update from Revision 19 dated 04/20/09.
- References to oil sample preparation have been removed.
- Extraction volumes for TCLP have been updated.

METALS DIGESTION/PREPARATION

References:

Methods 3005A, 3010A, 3050B and EPA 200.7

I. SCOPE AND APPLICATION

A. AQUEOUS

1. Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy".
 - a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
2. Method 200.7, "Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry".
 - a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
3. Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy".
 - a. This method is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for analysis by ICP. The procedure is used to determine total metals.

B. SOLIDS

1. Method 3050B, "Acid Digestion of Sediments, Sludges and Soils".
 - a. This method is used to prepare sediments, sludges and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.
 - b. It should be noted that some metals could be biased high with the soil digestion when dilution is necessary. Take necessary measures to ensure that dilutions are made as accurately as possible.

C. NOTES:

1. "Total Metals" includes all metals, inorganically and organically bound and both dissolved and particulate.
2. "Dissolved metals" includes all metals present in a sample after filtration through a 0.45 micron filter followed by digestion, when selenium is needed. The method does not require digestion for dissolved metals.

II. SUMMARY OF METHODS

- A. A representative sample of water or soil is put into an acid medium and exposed to heat for a certain amount of time. This allows for reduction of interferences by organic matter and converts metals bound to particulates to form the free metal that can be determined by ICP-Atomic Emission Spectrometry.

NOTE: When a reporting limit is required for a project lower than is customary, a four times concentration or alternate soil digestion ratio must be used in order to reach that lower level. Care must be taken to matrix match this concentrated aliquot. A blank and laboratory control sample (at a reduced concentration) are required with this concentration. A matrix spike (not at

reduced concentration) and duplicate or matrix spike and matrix spike duplicate is needed per 20 samples or per batch.

III. SAMPLE HANDLING AND PRESERVATION

A. AQUEOUS

1. Samples are taken in high density polyethylene, one liter bottles. Samples should be preserved with concentrated HNO₃ to a pH <2 immediately upon sampling. If dissolved metals are to be analyzed the sample should be filtered before the HNO₃ is added. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months. Note – samples received unpreserved and preserved in-house must be held 24 hours prior to preparation.

B. SOLIDS

1. Samples are taken in high density polyethylene or glass bottles. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

IV. INTERFERENCES

A. AQUEOUS

1. Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

B. SOLIDS

1. Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether this method is applicable to a given waste.

V. SAFETY

- A. Normal accepted laboratory safety practices should be followed while performing this analysis.
- B. Be certain the exhaust hood is functioning before you begin the digestion procedure.
- C. Hot acids can be extremely corrosive. Avoid inhalation or contact with skin.

VI. EQUIPMENT/APPARATUS

- A. Fume hood, Labconco or equivalent.
- B. Hot block, Environmental Express or equivalent source for use at 95°C. The temperature of the hot block must be monitored via the use of a temperature blank.
- C. Thermometer capable of reading 80 to 120 degrees C – ERTCO cat# 611-3-SC or equivalent.
- D. Vacuum pump for filtering dissolved metals- Gast or equivalent.
- E. Analytical balance capable of weighing to 0.01 gram. Mettler model BB300 or equivalent.
- F. Beckman CS-6R centrifuge.
- G. Various class A volumetric glassware, Pyrex or equivalent.
- H. Whatman No. 41 filter paper or equivalent.
- I. Whatman No. 42 filter paper or equivalent.
- J. Whatman 0.45 micron filter paper or equivalent.
- K. 250 mL beaker or other appropriate vessel such as polypropylene block digester tubes and caps.

- L. Stirring device, e.g. magnetic stirrer, glass rod or equivalent.
- M. Manual Sample Mill
- N. Wiley Sample Mill
- O. Clippers for cutting vegetation

NOTE: All glassware should be acid washed.

VII. REAGENTS AND STANDARD PREPARATION

A. REAGENTS

1. Metals grade Nitric acid (HNO_3). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
2. Metals grade Hydrochloric acid (HCl). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
3. 30% hydrogen peroxide reagent, ACS Grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
4. Reagent water (Deionized water).

B. STANDARDS

1. Traceability

- a. A LIMS record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the LIMS as well as on the container's label.
- b. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the information is recorded in LIMS. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in LIMS. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

2. PREPARATION

A. Blank Spike

1. Aqueous

- a. This solution is prepared as follows: 50 mL concentrated HCl , 20 mL concentrated HNO_3 , 1 mL of CLP-CAL-1, Solution A, 1 mL of CLP-CAL-1 Solution B, 0.25 mL of CLP-CAL-2, and 0.25 mL of CLP-CAL-3 diluted to 1 L in a volumetric flask. Use 50 mL for digestion. This solution is given a unique identifier and recorded in LIMS.
- b. For four times concentrated samples: The solution is prepared as follows: 50 mL concentrated HCl , 20 mL concentrated HNO_3 , 1 mL CLPP-SPK-4 (Inorganic Ventures) (This solution contains 10 mg/L Selenium, 100 mg/L Antimony, 50 mg/L Cadmium and Thallium, 40 mg/L Arsenic and 20 mg/L Lead) to 1 L in a volumetric flask. This solution is given a unique identifier. Use 12.5 mLs to 50 mLs and prepare two aliquots. Heat at 90 to 95°C to reduce the volume in each vessel to ten mLs and then combine each 10 mL aliquot into one vessel and take to a final volume of 25 mLs. Take care to

matrix match acids so that the final 25 mL portion will contain 2% HNO₃ and 5% HCl. Use 0.125 mLs HNO₃ and 0.3125 mLs HCl to each 50 mL vessel.

2. Solids:

- a. 1.0 ±0.02 (or 2.0 ±0.02) gram aliquot of teflon chips is weighed and spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier according to the Lot# for the teflon chips used and when digested is given the descriptor, i.e. BS1 and then BS2 etc. plus the unique identifier number assigned. Alternatively a solid matrix standard reference material is obtained from the manufacturer. This sample is given a unique identifier and the weight is recorded in a bound logbook and transferred to LIMS.

B. Spiking solution

1. Sample is spiked using 0.050 mL of CLP-CAL-1, Solution A and 0.050 mL of SM-391-001 for a final volume of 50 mL. These solutions are given unique identifiers. Use the circle sheet to indicate the amount spiked. Amount of spike used and the unique identifier of the standards are indicated in the bench sheets in Element as well.
2. For samples that require four times concentration, the sample is spiked using 0.025 mL of CLP-CAL-1, Solution A, 0.025 mL of CLP-CAL-1 Solution B, 0.0125 mL of CLP-CAL-2 and 0.0125 mL of CLP-CAL-3 for a final volume of 50 mL. When required, the sample is spiked using 0.025 mLs of B (1000 mg/L), 0.025 mL of Sn (1000 mg/L), 0.025 mLs of Mo (1000 mg/L), 0.025 mL of Titanium (1000 mg/L), 0.025 mL of Sr (1000 mg/L) and 0.025 mL of Li to a 100 mL vessel with 100 mLs of sample. The volume is lowered to less than 25 mLs and then final volume of this concentrated sample is brought back to 25mLs.
3. **For solid samples 1g to 200 mLs, the sample is spiked using 0.20 mL of CLP-CAL-1, Solution A and 0.20 mL of SM-391-001 for a final volume of 200 mL. These solutions are given unique identifiers. Use the circle sheet to indicate the amount spiked. Amount of spike used and the unique identifier of the standards are indicated in the bench sheets in Element as well.**
4. **For solid samples 2g to 100 mLs, the sample is spiked using 0.10 mL of CLP-CAL-1, Solution A and 0.10 mL of SM-391-001 for a final volume of 100 mL. These solutions are given unique identifiers. Use the circle sheet to indicate the amount spiked. Amount of spike used and the unique identifier of the standards are indicated in the bench sheets in Element as well.**

VIII. CALIBRATION

- A. The temperature of the samples must be maintained at 95°C and monitored via a temperature blank. Record in temperature logbook for later transfer into LIMS.

IX. PROCEDURE

- A. Calibrated digestion vessels are received from vendor with certificates indicating ASTM volume line calibration and lot #. Certificates are filed for future reference.
- B. Aqueous sample filtration (for dissolved metals):

1. Filter apparatus are purchased from a vendor; alternatively, thoroughly clean a flask and funnel with hot soapy water. Next, rinse the flask and funnel with 1:5 HNO₃ followed by a thorough D.I. water rinsing. This step is very important because the filters contain some metals (namely Zn) which could contaminate the samples.
 2. Rinse a 0.45 micron filter with 1:5 HNO₃ thoroughly, followed by D.I. water.
 3. Filter the unpreserved sample. If dissolved Hg analysis is requested for the sample, filter at least 200 mL.
 4. Discard the first 50 to 100 mL.
 5. A preparation blank must be taken through the filtration step and analyzed with the sample.
 6. Preserve the sample with HNO₃ to pH<2.
 7. Soluble samples that are clean and clear do not have to be digested. Use 100 mL sample, add 5 mL of concentrated HCl and 2 mL of concentrated HNO₃. **Samples must be digested unless approval for analysis without digestion is received from the project manager.**
- C. Aqueous sample preparation
1. Method 3005A "Acid digestion procedure for total recoverable or dissolved metals for analysis by ICP ", "Acid digestion procedure for total recoverable metals".
 - a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a digestion vessel. For samples which require concentration pour 50 mLs of the well-mixed sample into two digestion vessels.
 - b. Add 1.0 mL concentrated HNO₃ to the sample. For samples which require concentration, add 0.50 mL concentrated HNO₃ to the sample.
 - c. Add 2.5 mL concentrated HCl to the sample. For samples which require concentration, add 1.25 mL concentrated HCl to the sample.
 - d. Transfer the digestion vessel to a pre-heated hot plate at 90 to 95°C. A temperature blank will assure correct temperature. The temperature must be recorded in the temperature logbook. Take the volume down to between 5 to 10 mL, **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes.** Remove the sample from the hot plate and cool
 - e. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
 - f. Bring sample to its predigestion volume (or when samples require concentration, to a volume four times lower then what was started with) with DI water in the digestion vessel. The final volume must be recorded in the LIMS.
 - g. The sample is now ready for analysis.
 - h. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards plus identification #'s for standards used for spiking and the volume spiked into the sample.
 2. Method 200.7, "Acid digestion procedure for total recoverable or dissolved metals for analysis by ICP ", "Acid digestion procedure for total recoverable metals". If sample contains undissolved solids >1% refer to Section 11.3 of Method 200.7 for subsequent procedures.

- a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a digestion vessel. For samples which require concentration pour 50 mLs of the well-mixed sample into two digestion vessels.
 - b. Add 0.50 mL concentrated HNO_3 to the sample.
 - c. Add 0.25 mL concentrated HCl to the sample.
 - d. Transfer the digestion vessel to a pre-heated hot block at 95°C . A temperature blank will assure correct temperature. The temperature must be recorded in the temperature logbook. Take the volume down to between 5 to 10 mL, **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes.** Remove the sample from the hot block and cool
 - g. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
 - h. Bring sample to its predigestion volume with DI water in the digestion vessel. The final volume must be recorded in the LIMS.
 - g. The sample is now ready for analysis.
 - h. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards plus identification #'s for standards used for spiking and the volume spiked into the sample.
3. Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy".
- a. Shake sample thoroughly and pour 50 mL (5ml diluted to 50mL for TCLP, full 50ml volume for SPLP) of the well-mixed sample into the digestion vessel.
 - b. Add 1.5 mL concentrated HNO_3 to the sample.
 - c. d. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C . A temperature blank must be used, with the temperature being recorded in the temperature logbook. Take the volume down to a low volume (~5 mL), **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes. Also make certain that no portion of the bottom of the digestion vessel is allowed to go dry. This may lead to low recoveries.** Remove the sample from the hot plate and cool.
 - d. Add another 1.5 mL portion of concentrated HNO_3 to the sample.
 - e. Transfer the vessel to the hotblock or equivalent source. Increase the temperature so a gentle reflux occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).
 - f. Evaporate to a low volume (~3 mL) **making certain that no portion of the bottom of the digestion vessel is allowed to go dry.** Remove and cool.
 - g. Add 2.5 ml of concentrated HCl .
 - h. Reflux for an additional 15 minutes.
 - i. Bring sample to its predigestion volume in digestion vessel.
 - j. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

Note: When preparing DoD project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.

- k. The sample is now ready for analysis.
- l. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

D. Solid sample preparation

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

- *The material in the sample pan(inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

This procedure should be repeated several times until the sample is adequately mixed.

NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.

Grinding of Vegetation Samples

Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry enough where it won't stick to the inside of the mill. Grind the dried sample to fineness in either the manual sample mill or the Wiley mill or both if needed. Place the ground sample in a container and label immediately.

1. Method 3050B, "Acid digestion of Sediments, Sludges and Soils"
 - a. Mix the sample thoroughly for 5 minutes using a plastic spatula or Teflon coated spatula in a glass or plastic weigh boat to achieve homogeneity.
 - b. Weigh approximately (to the nearest 0.01 g) a 1 to 1.5 g portion of the sample directly into a digestion vessel. For samples with low percent solids a larger sample size may be used as long as digestion is completed. Record the exact mass in the LIMS.

NOTE: To achieve the lowest reporting limit possible, use a 2.0 g portion of sample with an ending volume of 100 mLs.

- c. Add 5 mL D.I. water and 5 mL concentrated $\text{HNO}_3(1:1)$, mix the slurry. Place the sample in a preheated hot block and reflux at 95°C for 10 to 15 minutes being certain that the sample does not boil. Record temperature in temperature logbook

- d. Allow the sample to cool. Add 5 mL concentrated HNO_3 , and heat/reflux again for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3 , repeat this step (addition of 5 mL of concentrated HNO_3) over and over until no brown fumes are given off by the sample indicating the complete reaction with HNO_3 . Using a watch glass or equivalent allow the solution to evaporate to approximately 5 mL without boiling at $95^\circ\text{C} \pm 5^\circ\text{C}$ for approximately two hours. Maintain a covering of solution over the bottom of the vessel at all times. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the beaker. If the volume does get low, add 2.5 mL of D.I. water to bring volume back up.
- e. Take the sample off the hot block and allow it to cool. Next, add 2 mL of D.I. water and 3 mL of 30% Hydrogen Peroxide. (The sample will bubble upon the addition of H_2O_2 if it is still warm.) Return the sample to the hot block or equivalent source and heat until the bubbling subsides. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker. Add two more 3 mL portions of H_2O_2 to the sample in the same manner as before. (NOTE: Do not add more than a total of 10 mL 30% H_2O_2 .)
- f. Continue heating the acid-peroxide digestate at $95^\circ\text{C} \pm 5^\circ\text{C}$ without boiling for approximately two hours until the volume has been reduced to approximately 2.5 mL. Maintain covering of solution over the bottom of the vessel at all times.
- g. Add 2.5 mL of DI water and 2.5 mL of concentrated HCl and 10 mL of DI water, cover the sample with a ribbed watch glass and continue refluxing for an additional 10 minutes without boiling
- h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
- i. Bring sample up to 50 mL with D.I. water in the vessel. Add 150 ml of DI water to a 250 ml sample bottle using a class A graduated cylinder. Invert the 50 ml sample digestion vessel several times to mix the sample and mix 50mL sample volume with the 150 mL in the sample bottle. Pour some sample back into the 50 ml sample digestion vessel to rinse and pour back into the 250 ml sample bottle and cap and mix.
- NOTE1:** When preparing DoD project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.
- NOTE2:** To achieve the lowest reporting limit possible use 2.0 grams of sample with an ending volume of 100 mLs.
- j. The sample is now ready for analysis.
- k. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

X. CALCULATIONS

- A. The analyst must record both beginning sample masses/volumes and final digestate volumes. This information must be recorded in the bench sheet.

XI. QUALITY CONTROL

A. Digestion

1. Temperature blank

- a. The temperature of the hot plate/hot block must be monitored for temperature during the digestion process.
- b. The thermometer must be tagged with annual calibration information. Record the thermometer reading, correction factor and the corrected temperature in the digestion log.

2. Blanks

- a. Digest a blank with every batch of samples digested (20 sample maximum). The blank is prepared by adding all the same reagents added to the samples to a clean dry beaker and taking it through the same process as the samples.
- b. Also, there must be a blank for every different method of digestion that is set up that day, every 20 samples.
- c. There must also be a blank for every different matrix of samples that is to be digested, every 20 samples.
- d. Sample is given a unique identifier in the digestion log.

3. Blank Spike

- a. For water samples, one BS is digested with every batch of samples digested (20 sample maximum).
- b. For water samples, a BS is digested every day for each type of digestion, every 20 samples.
- c. For soil/sediment samples, a soil matrix standard reference material (SRM) must be digested per batch (20 samples maximum) or alternatively a spiked teflon chip sample.
- d. Sample is given a unique identifier in the digestion log.

4. Duplicates

- a. A duplicate is prepared every 20 samples. This usually takes the form of a matrix spike duplicate.
NOTE: Certain projects require a sample duplicate and a matrix spike duplicate with each set of twenty samples.

B. Sample Matrix

NOTE: Field blanks/duplicates, trip blanks, or equipment blanks are not to be used for sample matrix QC samples.

1. Matrix spike

- a. Digest a spike and spike duplicate every 20 samples where sample volume is adequate to do so. Choose a sample (if possible) that has a lot of metals requested to be analyzed.

NOTE: For some projects, a sample duplicate and sample spike may be required instead of a spike and spike duplicate. Your supervisor should make you aware of these projects.

- b. The following metals do not get digested spikes when using CLP spike.

Calcium
Magnesium
Sodium
Potassium

- c. For TCLP samples, a spike **and a spike duplicate** must be digested for every matrix. You should inspect the sample (original sample prior to extraction) or check the log book to determine matrix type. (Also the matrix spike aliquots must be added to the extracts after filtration but before preservation.)
- d. **Certain projects require that a high and a low spike be prepared and analyzed. Check project specifications. Spikes should be prepared at 40 mg/Kg and 400 mg/Kg for soil samples and 200 ug/L and 2000 ug/L for aqueous samples.**

XII. CORRECTIVE ACTIONS

- A. Sample boils during digestion.
 - 1. Redigest another sample aliquot.
- B. Sample goes dry or portion of beaker bottom is exposed due to excess evaporation during digestion.
 - 1. Redigest another sample aliquot.
 - 2. Glass beaker dry for an extended period of time? Discard beaker.

XIII. SPECIAL NOTES

- A. **Never** take for granted how a sample should be digested. If the sample looks strange or unusual, or if you are not sure what metals the sample gets, what detection limits are required, whether the sample is total or dissolved, or even what method of digestion should be used, always ask your supervisor or the person who is to analyze the sample. How metals need to be digested changes too often to take it for granted.
- B. **Antimony (Sb) soils** should be analyzed within 48 hours of digestion whenever possible. When a soil requesting Antimony analysis is received, you must coordinate with the person who will be analyzing it to be sure that they can analyze it on the same day that it is digested.
- C. Labels for the digested sample must be written in a neat and legible manner. The labels must include such information as sample number, client name, the date digested, and the volume or mass digested.
- D. There are several precautions that must be taken to minimize the possibility of contamination.
 - 1. All metals glassware must be kept separate from all other laboratory glassware.
 - 2. Metals glassware must be washed as soon as possible after being used. **Dirty metals beakers must not be left overnight.**
 - 3. Acid to be used for metals digestions must be kept separate from all other laboratory acid.
- E. Samples must be digested in a timely manner to ensure ICP analysis remains on schedule for data generation. Samples received on or before Wednesday of week X must be prepared for ICP digestion by the end of week X. Your supervisor must be consulted if this schedule can not be met at a particular time.
- F. Please consult Waste Disposal SOP-QS14, for information concerning disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

XIV DEFINITIONS – Refer to SOP-QS08 for common environmental laboratory definitions.

XV ATTACHMENT – Circle Sheet for Eppendorfs and Solutions.

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ICP Metals: Circle Applicable Digestion/Spike and 0.1mL Eppendorf option then staple to bench sheet

Soil 2g-100mL (3050B)

Amount	BS/MS/MSD Standards 6010 Soil
0.10mL	#01 CLP-CAL-1 (50-33000ug/mL)
0.10mL	SM-391-001 (125->1000ug/mL)
	0.1ml Eppendorf 148897Z or 250847 (circle)
Amount	Reagents for Digestion 6010 Soil
5mL	HNO ₃
5mL	HNO ₃
2mL	DI H ₂ O
3mL	H ₂ O ₂
3mL	H ₂ O ₂
3mL	H ₂ O ₂
2.5mL	DI H ₂ O
2.5mL	Conc HCl
10mL	DI H ₂ O

Soil 1g-200mL (3050B)

Amount	BS/MS/MSD Standards 6010 Soil
0.20mL	#01 CLP-CAL-1 (50-33000ug/mL)
0.20mL	SM-391-001 (125->1000ug/mL)
	0.1ml Eppendorf 148897Z or 250847 (circle)
Amount	Reagents for Digestion 6010 Soil
5mL	HNO ₃
5mL	HNO ₃
2mL	DI H ₂ O
3mL	H ₂ O ₂
3mL	H ₂ O ₂
3mL	H ₂ O ₂
2.5mL	DI H ₂ O
2.5mL	Conc HCl
10mL	DI H ₂ O

TCLP 5mL-50mL (3010A)

Amount	MS/MSD Standards 6010 TCLP
5.0mL	Silver TCLP Spike
0.50mL	Main TCLP Spike
	1.0mL Eppendorf 1854694
	5mL Finnpiptette EH26207
Amount	BS Standards TCLP 6010
50mL	Water LCS (0.050-5.0ug/mL)
Amount	Reagents for Digestion TCLP
1.5mL	HNO ₃
1.5mL	HNO ₃
2.5mL	HCl

Water 100mL-25mL (3005A)

Amount	MS/MSD Standards 6010 Water
0.025mL	#01 CLP-CAL-1 (50-33000ug/mL)
0.025mL	#02 CAL-CAL-1 Solution B (250ug/mL)
0.0125mL	#03 CLP-CAL-2 (1000ug/mL)
0.0125mL	#04 CLP-CAL-3 (500ug/mL-1000ug/mL)
0.025mL	#05 Boron (1000ug/mL)
0.025mL	#06 Tin (1000ug/mL)
0.025mL	#07 Molybdenum (1000ug/mL)
0.025mL	#08 Titanium (1000ug/mL)
0.025mL	#09 Strontium (1000ug/mL)
0.025mL	#10 Lithium (1000ug/mL)
	0.1ml Eppendorf 148897Z or 250847 (circle)
Amount	BS Standards 6010 Water
2.0mL	Water LCS (0.050-5.0ug/mL)
0.5mL	Salt Spike (25ug/mL)
	1.0mL Eppendorf 1854694
	5mL Finnpiptette EH26207
Amount	Reagents for Digestion Low Water
0.5mL	HNO ₃
1.25mL	HCl

Water 50mL-50mL (3005A)

Amount	MS/MSD Standards 6010 Water
0.050mL	#01 CLP-CAL-1 (50-33000ug/mL)
0.050mL	SM-391-001 (125->1000ug/mL)
	0.1ml Eppendorf 148897Z or 250847 (circle)
Amount	BS Standards 6010 Water
50mL	Water LCS (0.050-5.0ug/mL)
Amount	Reagents for Digestion 6010 Water
1.0mL	HNO ₃
2.5mL	HCl

Water 50mL-50mL (200.7)

Amount	MS/MSD Standards 6010 Water
0.050mL	#01 CLP-CAL-1 (50-33000ug/mL)
0.050mL	SM-391-001 (125->1000ug/mL)
	0.1ml Eppendorf 148897Z or 250847 (circle)
Amount	BS Standards 6010 Water
50mL	Water LCS (0.050-5.0ug/mL)
Amount	Reagents for Digestion 6010 Water
0.50mL	HNO ₃
0.25mL	HCl

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

METALS: SOP 105

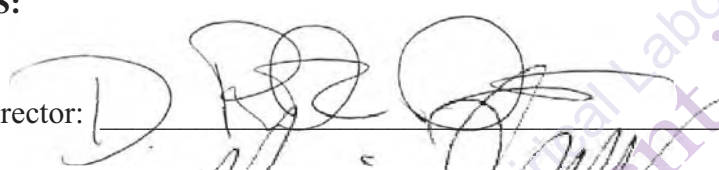
REVISION #: 20

EFFECTIVE DATE: 20130701

**METALS
BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION
SPECTROMETRY (ICP-AES) TECHNIQUE
SW846 6010B, SW846 6010C, EPA 200.7 and SM 2340 B-2011 for Hardness Calculation**

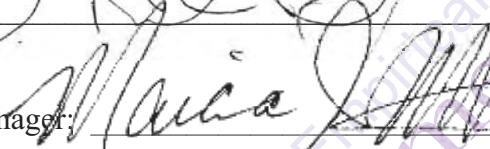
APPROVALS:

Lab Director:



Date: 07/01/2013

Data Quality Manager:



Date: 07/01/2013

Section Supervisor:



Date: 07/01/2013

Changes Summary

Revision 20, 20130701

- Updated standard methods reference to reflect SM 2340 B-2011 rather than SM 2340B (19th, 20th and 21st Edition).
- Removed CLP references/procedures throughout SOP.
- Standard stock concentrations corrected for Aluminum, Calcium, Iron and Magnesium in section 10.6.3.
- RL standard clarified in section 12.1.5 to indicate also known as LLICV/LLCCV with DoD QSM limits on ICV of $\pm 20\%$ but 6010C limits of $\pm 30\%$.
- Serial Dilution in section 12.1.9 updated to indicate a 5x dilution instead of 1:4 as in 6010A or 1:5 as in 6010B/6010C.
- Updated section 12.3.2 to be consistent with QC table 2.
- Reference to USACE removed from section 14.2 and 14.4.
- LLICV and LLCCV added to QC table 2.

Revision 19, 20120813

- Software qualifier definitions added Table 1C.
- Analyst review checklist updated
- Table of metals added listing all the metals affected when another metal is present Table 1B.

Revision 18, 20120720

- Archival/PDF access of standard COAs and reagent checks added to section 10.
- Standard expiration dates addressed in section 10.
- Appendix I information added as referenced in section 10.
- Standard methods references added to section 21.
- Cadmium wavelength update in table 1A.
- Table 1 updated to reflect MDL/DL, LOD and LOQ/MRL for all metals currently calibrated.

Revision 17, 20110516

- This is an update of SOP revision 16 dated 4/11/2010.
- Change all limit statements to include “after rounding to the nearest whole number”.
- Add procedure for recording digestates filtered prior to analysis within section 14.2.
- Training SOP reference updated to QS03 in section 14.6.
- References to DoD QSM 4.1 have been updated to DoD QSM 4.2.

Revision 16, 04/11/10

- The SOP is an update from Revision 15 dated 05/08/09
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory’s revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
3. Detection Limit
4. Scope of Application, Including components to be Analyzed
5. Summary of the Test Method
6. Definitions
7. Interferences
8. Safety
9. Equipment & Supplies
10. Reagents and Standards
11. Sample Collection, Preservation, Shipment, and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Data Analysis and Calculations
16. Method Performance
17. Pollution Prevention
18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Contingencies for Handling out-of-control or unacceptable data
20. Waste Management
21. References
22. Tables, Diagrams, Flowcharts and Validation Data

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1. Identification of the Test Method

This SOP is compliant with methods – SW846 6010B, SW846 6010C, EPA 200.7, and SM 2340 B-2011 for Hardness Calculation.

2. Applicable Matrix or Matrices

This SOP is applicable to all matrices, including ground water, aqueous samples, TCLP, SPLP and EP extracts, industrial and organic wastes, soils, sludge samples, sediments, and other solid wastes, require digestion prior to analysis.

3. Detection Limit: Detection limits are found in Table 1 of this SOP. Sensitivity and optimum ranges of the metals may be found in the ICP method file.

4. Scope of Application, Including components to be Analyzed

Each parameter that is analyzed and reported under the scope of this SOP is listed in Table 1 of this SOP. This table also lists the associated Method Detection Limit and the Reporting Limit (also defined as the Limit of Quantitation).

5. Summary of the Test Method

5.1 Prior to analysis, samples must be solubilized or digested using appropriate sample Preparation Methods (e.g., Methods 3005-3050). When analyzing for dissolved constituents, acid digestion is not always necessary if the samples are filtered and acid preserved prior to analysis. If particulates form after filtration and preservation the sample must be digested prior to analysis.

NOTE: When selenium is required soluble samples must always be digested.

5.2 This method describes the simultaneous multi-elemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the large droplets are removed by a spray chamber and the small droplets then pass through to the plasma. The solvent is evaporated. The residual sample decomposed to atoms and ions that become excited and emit characteristic light which is measured, giving a measurement of the concentration of each element type in the original sample. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analytic wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. Control of the spectrometer is provided by PC based *iTEVA* software.

5.3 Inductively Coupled Argon Plasma (ICAP) primary advantage is that it allows simultaneous determination of any elements in a short time. The primary disadvantage of ICP is background radiation from other elements and the plasma gases. Although all ICP instruments utilize high-resolution optics and background correction to minimize these interferences, analysis for traces of metals in the presence of a large excess of a single metal is difficult. Examples would be traces of metals in an alloy or traces of metals in a limed (high calcium) waste. ICP and Flame AA have comparable detection limits (within a factor of 4) except that ICP exhibits greater sensitivity for refractories (Al, Ba, etc.). Furnace AA, in general, will exhibit lower detection limits than either ICP or FAA.

5.4 It is standard procedure to use an internal standard (scandium) with samples to increase the stability of the instrument as recommended by the manufacturer (Thermo Fisher). (When samples are suspected of containing scandium, internal standard cannot be used.)

6. Definitions

Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

Additional definitions specific to this SOP are listed below:

- 6.1 **ICP or ICAP**- Inductively Coupled Plasma or Inductively Coupled Argon Plasma.
- 6.2 **Inter-element correction (IEC)**- Defined as a correction factor applied by the instrument when there is an overlap of the spectrum from the plasma gases or from another metal into the spectrum of another metal causing that metals concentration to either be inflated or deflated.

7. Interferences

- 7.1. Spectral interferences are caused by background contribution from continuum or recombination phenomena, stray light from the line emission of high-concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.
 - 7.1.1. Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be included in the correction algorithm. Off-line interferences are handled by including spectra on interfering species in the algorithm.
 - 7.1.2. To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line inter-element spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a 200 mg/L or 500 mg/L concentration near the upper analytical range limit.

- 7.1.3. Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for inter-element contributions. Instruments that use equations for inter-element correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply inter-element correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelength are listed in the method in table 2. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.
- 7.1.4. When using inter-element correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that Arsenic is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Aluminum. According to Table 2 from the method, 100 mg/L of Aluminum would yield a false signal for Arsenic equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Aluminum would result in a false signal for Arsenic equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interferences than that shown in Table 2 from the method. The interference effects must be evaluated for each individual instrument since the intensities will vary.
- 7.1.5. Inter-element corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences.
- 7.1.6. The interference effects must be evaluated for each individual instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences as well as any other suspected interferences that may be specific to the instrument or matrix. The instrument utilizes a computer routine for automatic correction on all analyses.
- 7.1.7. If the correction routine is operating properly, the determined, apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and

corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

- 7.1.8. When inter-element corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions (IFA/IFB). If the correction factors or multivariate correction matrices tested on a daily basis are found to be within 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis. Also, if the nature of the samples analyzed is such they do not contain concentrations of the interfering elements at \pm one reporting limit from zero, daily verification is not required. All inter-element spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation-change, such as in the torch, nebulizer, injector, or plasma conditions occurs. Standard solution should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.
- 7.2. Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers.
- 7.3. Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build-up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the elements and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized.
- 7.4. Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. When the instrument displays negative values, dilution of the samples may be necessary.

8. Safety

Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab-wide.

- 8.1 Normal accepted laboratory safety practices should be followed while performing this analysis.
 - 8.1.1. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of appropriate safety gloves and lab coats is highly recommended.
 - 8.1.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
 - 8.1.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves in the Data Quality Managers office.

9. Equipment & Supplies

- 9.1. Inductively coupled argon plasma emission spectrometer: Thermo Scientific 6500 DUO.
- 9.2. Computer-controlled emission spectrometer with background correction: Thermo Scientific 6500 DUO or equivalent.
- 9.3. Radio frequency generator compliant with FCC regulations: Thermo Fisher or equivalent.
- 9.4. Auto-sampler: Thermo Fisher or equivalent.
- 9.5. Printer capable of printing results every 4 minutes.
- 9.6. Cooling Water recycler.
- 9.7. Iteva software.
- 9.8. Argon gas supply – Liquid Argon
- 9.9. Class A volumetric flasks
- 9.10. Analytical balance - capable of accurate measurement to a minimum of three significant figures (0.001gm).
- 9.11. Variable Eppendorf Pipettes 1000µL; 5000µL
- 9.12. Disposable beakers 10, 20 and 50 mL size.
- 9.13. Hood system capable of venting the heat from the system off of the instrument during analysis.

10. Reagents and Standards

The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

Note: Unless specifically indicated below or vendor/source standard indicates earlier expiration date, stock standards and reagents are given 12 month expiration dates, digestion spike standards are given 6 month expiration dates and working calibration/IEC standards are given 3 month expiration dates.

- 10.1. Reagent Water. All references to water in the method refer to reagent grade water unless otherwise specified. Reagent water will be interference free.
- 10.2. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL/DL, then the reagent is acceptable. All reagents must be logged into Element and assigned an ID.

- 10.3. Hydrochloric acid (concentrated), HCl. A method blank is digested and analyzed before a new lot number of HCl is put into use, to ascertain purity. The lot # is logged into Element and the data forwarded to the administrative department with Element ID indicated so PDF can be generated and associated for access within Element and appropriate archival
- 10.4. Nitric acid (concentrated), HNO₃. A method blank is digested and analyzed before a new lot number of HNO₃ is put into use, to ascertain purity. The lot # is logged into Element and the data forwarded to the administrative department with Element ID indicated so PDF can be generated and associated for access within Element and appropriate archival.
- 10.5. Calibration standards – Purchased standards must be received with a Certificate of Analysis (COA). The standard must be logged into the LIMS to receive an ID which is then written on the COA. The COAs are then forwarded to the administrative department with ID indicated so PDF can be generated and associated for access within Element and appropriate archival.
- 10.5.1. All standards have an acid matrix of 2% HNO₃ and 5% HCl and should be prepared using class A volumetric flasks and calibrated Eppendorfs).
- 10.5.2. CAL1 is the calibration blank: Reagent grade water matrix matched as in 10.5.1.
Note: when this standard is analyzed the intensities should be compared to a previous run to make sure that no contamination has occurred. Prepare this solution fresh daily.
- 10.5.3. Stock QC21 solution: (100 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals - Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, Se, Sr, Tl, Ti, V, and Zn.
- 10.5.4. Stock QC7 solution: Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals- (50 ug/mL)- silver; (100 ug/mL)- aluminum, boron, barium and sodium; (1000 ug/mL)- potassium; (500 ug/mL or 100 ug/mL note we use two sources of this standard and each have different concentrations for Si) –Silica.
- 10.5.5. Boron solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.6. Stock Tin solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.7. Stock Silver solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.8. Stock Aluminum solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.9. Stock Calcium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier. Note: Two sources are needed.
- 10.5.10. Stock Magnesium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

- 10.5.11. Stock Iron solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.12. Stock Potassium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.13. Stock Barium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.14. Stock Sodium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.15. Stock Arsenic solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.16. Stock Cobalt solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.17. Stock Chromium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.18. Stock Copper solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.19. Stock Manganese solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.20. Stock Nickel solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.21. Stock Lead solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.22. Stock Selenium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.23. Stock Thallium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.24. Stock Beryllium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.25. Stock Cadmium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.26. Stock Antimony solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

- 10.5.27. Stock Molybdenum solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.28. Stock Strontium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.29. Stock Titanium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.30. Stock Vanadium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.31. Stock Zinc solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.32. Stock Scandium solution (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.6. Calibration and Calibration Verification standards

- 10.6.1. The calibration standards and calibration verification standards preparations are recorded in Element. Please find method of preparation in Appendix I. ICV and CCV standards are prepared daily.
- 10.6.2. The CRL solution is analyzed to check the accuracy of the instrument at the reporting limit. The stock standard solutions A and B are prepared from single element standards listed in 10.5 above. Please find method of preparation in Appendix I. Prepared by adding 1.0 ml of RL Stock solution A and 1.0 ml of RL Stock Solution B to de-ionized water with 2% HNO₃ and 5% HCL matrix and diluting to 100 mLs , mix well.
- 10.6.3. The interference check standard solutions (IFA and IFB) are prepared to provide an adequate test of the IECs. A purchased solution containing 5000 ug/mL Al, Ca, Mg and 2000 ug/mL Fe is diluted 10x to prepare the IFA. The IFB is prepared by diluting 100x a purchased solution containing 10 ug/mL of As and Tl; 20 ug/mL Ag; 50 ug/mL Ba, Be, Cr, Co, Cu, Mn, and V; 100 ug/mL Cd, Ni and Zn; 5 ug/mL Pb and Se; and 60 ug/L Sb. Add to this a purchased solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe diluted 10x.

10.7 Digestion standards

- 10.7.1 The Blank Spike (BS) is prepared from High Purity solutions CLP-CAL-1 solution A and B; CLP-CAL-2 and CLP-CAL-3. 0.50 mL of CLP-CAL-1 A and B; and 0.50 mLs of the 1000 ug/mL single element standards for Molybdenum, Boron, Titanium and Strontium is diluted to 500 mL with 0.125 mL of CLP-CAL-2 and CLP-CAL-3 and 0.050 mLs of 10000 ug/mL Tin. 25 mL of HCl and 10 mL of HNO₃ are added for preservation. This solution is stored in a Teflon bottle. A portion is reserved in case of a problem with digestion. When there is a problem with the analysis of the BS the solution is checked first before action is taken to make sure that it was made properly and has not deteriorated since it was made up. This solution is given a unique identifier within

Element. The BS is prepared from a source independent from that used in the calibration standards. 50 mLs of this solution is used for digestion for normal level water samples and the sample is brought back to 50 mLs after digestion. Low level water samples start with two 50 mLs vials with only 1.0 mL of the stock blank spike solution in each taken to 50 mLs. The samples are cooked down to below 25 mLs and combined and then cooked down to below 25 mLs again and then brought back to 25 mLs. This low level BS is given a unique identifier in Element.

10.7.2. The solid BS used with soil samples is prepared by weighing up 1.0 gram of Teflon chips for regular level and 2.0 grams of Teflon chips for low level and spiking using the same spiking solutions used to spike the sample matrix. This standard is given a unique identifier i.e. Batch #-BS1. Note: Amount of spiking solution used varies according to whether the samples are being digested for normal level or low level soils. See spiking solutions in 10.7.3.1 for how to prepare the BS for a solid sample, it is prepared the same way that a soil spike is prepared only the known amounts of metals are added to laboratory water.

10.7.3. The spiking solutions are prepared as follows:

10.7.3.1. Stock Multi-element Spiking Solutions: High Purity CLP-CAL-1 solution A: 2000 ug/mL Al and Ba; 50 ug/mL Be; 200 ug/mL Cr; 500 ug/mL Co, Mn, Ni, V and Zn; 250 ug/mL Cu; 1000 ug/mL Fe; 5000 ug/mL Ca, Mg, K and Na; solution B: 250 ug/mL Ag; CLP-CAL-2: 1000 ug/L Sb; CLP-CAL-3: 1000 ug/mL As, Pb, Se, Tl; 500 ug/mL Cd. Order from the manufacturer already prepared. These solutions are given a unique identifier within Element. Add 0.050 mL for water samples and 0.20 mL for normal level soil samples and 0.10 for low level soil samples of CLP-CAL-1 solutions A and B, and 0.0125 mL for water samples and 0.05 mLs for normal level soil samples and 0.025 mLs for low level soil samples of CLP-CAL-2 and 3 to 50 mL of sample for water samples and 1 gram of sample for normal level soils and 2 grams of sample for low level soils for the following spike values: 2000 ug/L Al and Ba; 50 ug/L Be; 200 ug/L Cr; 500 ug/L Co, Mn, Ni, V and Zn; 250 ug/L Cu; 1000 ug/L Fe; 5.0 mg/L Ca, Mg, K and Na, 250 ug/L Ag, Sb, As, Pb, Se and Tl; 125 ug/L Cd. A blank spike should be prepared at the time the samples are spiked to check the actual spike value and accuracy.

10.7.3.2. TCLP Spiking Solution: Use 0.50 mL diluted to 50 mL for digestion: 2.5 mL 10000 mg/L Ba stock standard diluted to 100 mL; 2.5 mL Cr, Pb and As 1000 mg/L stock standard diluted to 100 mL; 0.50 mL Cd and Se diluted to 100 mL. Store in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 2500 ug/L Ba; 250 ug/L Cr, Pb and As; and 50 ug/L of Cd and Se. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed.

10.7.3.3. TCLP Silver Spiking Solution: Use 5.0 mL diluted to 50 mL for digestion: 0.40 mL of 1000 mg/L stock Ag solution diluted to 200 mL. Store this solution in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 200 ug/L. Note: Since the samples are diluted 10x when digested the spike value will

appear to be 10x greater when analyzed. This solution is assigned a 3 month expiration date.

11. Sample Collection, Preservation, Shipment, and Storage

Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

- 11.1. Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been pre-filtered and acidified will not need acid digestion as long as the samples and standards are matrix matched and particulates do not form after the filtration and preservation take place. Solubilization and digestion procedures are presented in Sample Preparation Methods (Methods 3005A-3050A).
- 11.2. Sample digestates are stored at room temperature for at least 3 months unless a longer time is requested by the client. The samples contain an acid matrix of 3:1. All metal samples are neutralized before disposal in the receiving section of the laboratory.
- 11.3. **The appropriate SOPs should be consulted regarding sample preparation.** The following is a brief summary of the methods we use for metals preparation.
 - 11.3.1. Method 3005A prepares groundwater and surface water samples for total recoverable and dissolved metals determination by ICP. The unfiltered or filtered sample is heated with dilute HCl and HNO₃ prior to metal determination.
 - 11.3.2. Method 3010A prepares waste samples for total metal determination by ICP. The samples are vigorously digested with a mixture of nitric acid and hydrochloric acid followed by dilution with laboratory water. The method is applicable to aqueous samples, TCLP and mobility-procedure extracts.
 - 11.3.3. Standard Methods Method 3030C prepares ground-waters and surface water samples for acid extractable metals: (lead and chromium.) This preparation has a holding time of 72 hours. The samples are preserved at collection with 5mL/L of HNO₃, in the laboratory 5 mL/100mL of 1+1 HCl is added and the sample is heated for 15 minutes in a block digester. Once cooled, the sample is filtered through a membrane filter and the filtrate is carefully transferred to a volumetric flask and brought back to 100 mLs.
 - 11.3.4. Method 3050B prepares wastes samples for total metals determination by ICP. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either laboratory water or hydrochloric acid and laboratory water. The method is applicable to soils, sludges, and solid waste samples.

12. Quality Control

Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.1. Daily run and batch QC

12.1.1. Calibration is required daily. Either a blank and a high standard or a client specific three standard concentration points and a blank calibration is required daily.

12.1.2. IEC correction standards for aluminum and iron are required daily.

12.1.3. ICV within $\pm 5\%$ for 200.7 and within $\pm 10\%$ for all other methods.

12.1.4. ICB/CCB less than two times \pm MDL/DL or less than \pm LOD for DOD. The ICB/CCB must immediately follow the ICV/CCV.

12.1.5. RL standard - also known as low-level ICV or CCV (LLICV or LLCCV) - analyzed against the curve within $\pm 20\%$ initially ($\pm 20\%$ DoD QSM, $\pm 30\%$ 6010C) and $\pm 30\%$ at the end of sample analysis.

12.1.6. IFA/IFB analyzed daily. IFA must be less than two times \pm MDL/DL or less than \pm LOD unless verified standard contamination for DOD. The IFB must recover within $\pm 20\%$ for all analytes in the IFB standard solution. If the IFA/IFB solution is not within the required limits- if possible reanalyze all associated samples, if not possible to reanalyze all associated samples must be qualified with a "Q" on the final report for DOD.

12.1.7. CCV must be analyzed every ten field samples and at the end of the analysis within $\pm 10\%$ or the samples are reanalyzed if possible. If samples cannot be reanalyzed, all samples are qualified with a "X" (high) or "Y" (low) for DOD.

12.1.8. CCB must be analyzed every ten samples immediately following the CCV or at the end of the analysis less than two times \pm MDL or $\leq \pm$ LOD for DOD. If the CCB is out of the allowable range the samples are qualified with "B".

12.1.9. *The following should be analyzed with each preparation batch containing a matrix spike.*

- Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 50 above the instrumental detection limit after dilution), an analysis of a 5x dilution (volumetric glassware must be used) should agree within $\pm 10\%$ of the original determination. If not, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.
- Post digestion spike addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value for DoD samples/SW6010B and 80 to 120% for SW6010C and is required especially if the pre-digestion matrix spike is outside of control limits. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected. Run all associated samples in the preparatory batch by method of standard additions (MSA) or apply "J" qualifier. The analyst and or section manager must note this situation on the final analytical report. Apply "J" qualifier if the post spike is outside the range of 75% to 125% for DoD/6010B or 80% to 120% for non-DoD 6010C.

12.2 Quarterly and/or every six months

- 12.2.1. Linear range standards must be analyzed at a frequency no less than once every six months. The linear range standard is required for verification that samples are actually linear to the degree claimed. The analyst is responsible for completing this task in a timely manner. The linear range standard must be within +/-10% of true value. This standard can be analyzed as the linear dynamic range.
- 12.2.2. The inter-element correction factors (IEC) should be verified at the time the linear range standards are analyzed or whenever there is any question about whether an IEC is correcting correctly.

12.3. Digested Batch QC

- 12.3.1. All quality control data should be maintained and available for easy reference or inspection.
- 12.3.2. Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank (BLK), sometimes referred to as the preparation blank, is a volume of reagent water acidified with the same amounts of acids as were the standards and samples. These blanks are taken through the same digestion/preparation steps as the sample being tested. The result for the method blank should not indicate contamination greater than $\frac{1}{2} \pm \text{RL}$ (project reporting limit) for DoD, and $< \pm 2 \times \text{MDL}$ for all others. If exceeded, the impact upon the data should be evaluated and the associated sample(s) should be either re-digested or the data should be qualified. The extracted blank associated with TCLP batches must be less than 100 X the regulatory limit for barium.
- 12.3.3. Employ a minimum of one blank spike (BS) for aqueous samples or one Teflon chip spiked sample per sample batch to verify the digestion procedure. These blank spikes are taken through the same digestion/preparation steps as the sample being tested. The control limits are $\pm 15\%$ method 200.7 - aqueous and soil samples or $\pm 20\%$ for all other methods aqueous and soil samples. If the BS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be re-digested. Consult your supervisor for further action. Qualifying the associated data may not be permissible for some clients.

12.4. Sample

- 12.4.1. Analyze one replicate sample for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. Project specific requirements will take precedence in these situations. NJDEP demands that this requirement be met with a client specific duplicate rather than a spike duplicate. The control limits are less than or equal to 20% RPD (if both are $> 5 \times \text{RL}$) or \pm the RL (if either are $< 5 \times \text{RL}$). Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. Apply "*" qualifier for all work if acceptance criteria are not met.
- 12.4.2. Analyze a minimum of one spiked sample and/or spiked sample duplicate for every twenty samples or per analytical batch, whichever is more frequent. Project specific requirements will take precedence in determining whether a matrix spike duplicate is employed in these situations. If the analyte level in the sample is not greater than 4X the spiking level, the spike recoveries should be within $\pm 20\%$ of the true value. If not, and sufficient sample volume

exist, a post digestion spike should be analyzed. Apply “N” qualifier for all work if acceptance criteria are not met.

13. Calibration and Standardization

Quality Systems **SOP QS08** “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

- 13.1. Set up the instrument with proper operating parameters. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).
- 13.2. Operating conditions - **The instrument settings can be found in method file within the iTEVA software.** For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.
- 13.3. Auto-peak when some change has been made to the introductory system and calibrate the instrument according to the instrument manufacturers recommended procedures, using the specified calibration standard solutions. Flush the system with 2% HNO₃ / 5% HCl between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a blank and three standards ($r \geq 0.998$). If a three point calibration curve is not required for the client samples being analyzed by Empirical Laboratories may use a blank and one standard as referenced in the method.
- 13.4. Before beginning the sample run, analyze single element Iron and Aluminum standards at their linear range to check for IEC drifts. Analyze these standards first as QC samples with an IEC check table and action taken should be to calculate IECs using the iTEVA software. Make sure to rinse thoroughly after running these linear range standards, they can cause carry over into the initial QC samples which are analyzed next. The analysis order follows as: ICV $\pm 10\%$ (for 200.7 - $\pm 5\%$) and ICB ($< \pm 2 \times \text{MDL/DL}$, $< \pm \text{LOD-DOD}$ or $\pm \text{RL/CRDL}$ for others), first, then analyze a reporting limit standard (a standard at the concentration of the reporting limit). This standard must be within $\pm 20\%$ for DOD projects and within $\pm 30\%$ for samples analyzed for 6010C. Note: Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.
- 13.5. Verify the inter-element and background correction factors at the beginning of the sequence in the specific order of IFA, IFB, CCV and CCB (IFA criteria: non-spiked analytes $< \pm 2 \times \text{MDL}$ or $< \pm \text{LOD}$ for DOD beginning of sequence. Do this by analyzing the interference check solution IFA and IFB. Absolute value of concentration for all non-spiked analytes in the IFA must be $< \text{LOD}$ (unless they are verified trace impurity from one of the spiked analytes) for DOD. Results must be within $\pm 20\%$ of the true value for IFB. If corrective action fails, apply Q-qualifier to all results for specific analyte(s) in all samples associated with the ICS.

Note: Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.

13.6. The instrument must be calibrated once every 24 hours.

13.7. Instrument Autosampler Report example:

Calibration Rack (used by instrument software to insert QC)

- 1) Cal Std 1 (blank)
- 2) Cal Std 2 (Low Cal)
- 3) Cal Std 3 (Mid Cal)
- 4) Cal Std 4 (Ba @ 5000 ppb, Ag @ 2000 ppb, Mn @ 10,000 ppb)
- 5) Cal Std 5 (QC5)
- 6) Cal Std 6 (QC 21)
- 7) Cal Std 7 (NAK 100)
- 8) Cal Std 8 (QC3)
- 9) Al IEC-(correction using ITEVA software)
- 10) Fe IEC-(correction using ITEVA software)

Sample Sequence RACK 1

- 1) SEQ-ICV
- 2) SEQ-ICB
- 3) SEQ-CRL1-reporting limit standard 1
- 4) SEQ-CRL2-reporting limit standard 2
- 5) Rinse
- 6) SEQ-IFA1
- 7) SEQ-IFB1
- 8) Rinse
- 9) SEQ-CCV
- 10) SEQ-CCB
- 11) Method Blank (*Batch # -BLK1*)
- 12) Blank Spike (*Batch # -BS1*)
- 13) Sample 1
- 14) Sample 2
- 15) Sample 3
- 16) Sample 4
- 17) Sample 5
- 18) Sample 6
- 19) Sample 7
- 20) Sample 8
- 21) Sample 9
- 22) Sample 10
- 23) SEQ-CCV
- 24) SEQ-CCB
- 25) Sample 11
- 26) Sample 12

- 27) Sample 13
- 28) Sample 14
- 29) Sample 15
- 30) Sample 16
- 31) Sample 17
- 32) Sample 18
- 33) Sample 19
- 34) Sample 20
- 35) Sample matrix spike (*batch#*- MS1)
- 36) Sample matrix spike duplicate (*batch#* -MSD1)
- 37) Sample post digestion spike (*batch#* -PS1)
- 38) Sample serial dilution (*batch#* -DUP1)
- 39) SEQ-CCV
- 40) SEQ-CCB
- 41) Preparation Blank (*batch#* -BLK1)
- 42) Blank Spike (*batch#* -BS1)
- 43) Sample 1
- 44) Sample 2
- 45) Sample 3
- 46) Sample 4
- 47) Sample 5
- 48) Sample 6
- 49) Sample 7
- 50) Sample 8
- 51) Sample 9
- 52) Sample10
- 53) SEQ-CCV
- 54) SEQ-CCB
- 55) Sample 11

RACK 2

- 1) Sample 12
- 2) Sample 13
- Etcetera...

Each rack holds 60 samples and there are 4 racks that are used for samples, CCVs and CCBs and run QC.

14. Procedure

14.1. Once the instrument has been calibrated, begin the analysis of samples.

14.2. If particulates are visible in the digestate, the sample must be filtered prior to analysis. If filtration is required, a filter blank must be prepared by filtering reagent grade water which has been properly acidified. The sample requiring filtration must be recorded on the bench sheet and added to the bench sheet comments in the LIMS.

14.3. Flush the system with 2% HNO₃ / 5% HCl for at least 1 minute before the analysis of each sample.

- 14.4. Dilute and reanalyze samples that are more concentrated than the linear calibration limit or, for 200.7, $\pm 10\%$ of the linear range standard.
- 14.5. Verify calibration every 10 field samples or every 2 hours, whichever is more frequent and at the end of the analytical run, using continuing calibration verification (CCV) sample and a continuing calibration blank (CCB) sample.
- 14.5.1. The results of the CCV are to agree within $\pm 10\%$ for 6010 (5% for 200.7) on initial verification of the expected value, with relative standard deviation (RSD) $< 5\%$ from 3 replicates (minimum of three integrations). If not, terminate the analysis, correct the problem, and reanalyze the previous ten samples. The analyst **may** continue the analytical run, and after conferring with the section manager it **may** be necessary to reanalyze a group of samples. The analyst must notify the section manager within 24 hours and prepare a non-conformance report as indicated.
- 14.5.2. The results of the calibration blank (this is not the method/preparation blank) are to be $< 2x \pm MDL$, for **DOD no analytes detected $> 1/2$ the project reporting limit**. If the calibration blank is not in control, evaluate the impact upon the previous 10 samples. Reanalysis may be required after an evaluation of the data. If the blank $< 1/10$ the concentration of the action level of interest and no sample is within 10% of the action limit, samples need not be reanalyzed. One must also evaluate the reporting limit (RL) as it relates to 3X the IDL/MDL. If the RL is significantly above 3X IDL or MDL then reanalysis may not be required (Na, K, Mg and Ca are good examples of this situation).
- 14.6. Demonstration of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to section 16 of this SOP.

15. Data Analysis and Calculations

Quality Systems SOP QS09 “General and commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

- 15.1. Total hardness is reported from HNO_3 preserved sample. The final concentration is calculated from the calcium and magnesium results as follows: $\text{Ca mg/L} \times 2.5 + \text{Mg mg/L} \times 4.1 = \text{total Hardness in mg/L as CaCO}_3$.
- 15.2. The instrument will generate data results in mg/L or $\mu\text{g/L}$ (labeled appropriately). Each result represents an average of three individual readings per metal channel.
- 15.3. For aqueous samples, if a post/pre-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution.
- 15.4. For solid samples, if a post-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution. Also, the result must be converted to reporting units which are usually mg/kg.

$$SR \text{ (ug/g or mg/kg)} = IR * DF * FED / SM$$

SR = Sample result
 IR = Instrument result (µg/L)
 DF = Dilution factor (post digestion)
 FED = Final volume of digestate (L)
 SM = Sample mass digested (g)

16. Method Performance

Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality Manual is completed by each analyst and then provided to the supervisor for further processing and approval.

17. Pollution Prevention:

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, “Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results”, provides details on data assessment and acceptance criteria for Quality Control Measures. Table 2 of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, “Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results”, provides details on handling out of control data. Table 2 within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

CORRECTIVE ACTIONS FOR NON-CONFORMANCES

19.1. INSTRUMENT RELATED

19.1.1.ICV

- a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
- b. Is the problem with the calibration?
 - i. Recalibrate through analysis of appropriate standards and recheck ICV.

19.1.2.ICB

- a. Is the problem with the solution?

- i. Re-prepare
- b. Is the problem with the calibration?
 - i. Recalibrate with the blank solution or the low level standard. Restart analysis with the ICV.

19.1.3. Check standards

- a. Is the problem with the solution?
 - i. Re-pour, re-prepare or obtain new stock.
- b. Is the problem with the calibration?
 - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.4. IFA

- a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
- b. Is the problem with the calibration?
 - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.5. IFB

- a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
- b. Is the problem with the calibration?
 - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.6. CCV

- a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
- b. Is the problem with the calibration?
 - i. If appropriate, continue the analysis. Discuss effect of the out of control situation with your supervisor. The samples will be reanalyzed or the data will be qualified.

19.1.7. CCB

- a. Is the problem with the solution?
 - i. Re-prepare
- b. Is the problem with the calibration?
 - i. Re-calibrate and reanalyze.

19.2. DIGESTION RELATED

19.2.1. Preparation blank (BLK)

- a. Is the problem with the instrument?
 - i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
- b. Is the problem with the digestion?
 - i. If associated samples are less than 10X the level of the preparation blank but above the RL, the sample must be re-digested or the data must be qualified on the final report.

19.2.2. BS

- a. Is the problem with the instrument?
 - i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
- b. Is the problem with the digestion?
 - i. If biased low, associated samples must be re-digested.

- ii. If biased high, the impact upon the data user must be evaluated. The samples will be re-digested or the data will be qualified on the final report.

19.3. SAMPLE MATRIX RELATED

19.3.1. Replicate analysis RPD

- a. The associated sample data must be qualified on the final report.

19.3.2. Spike analysis recovery

- a. Is the analyte level in the sample greater than 4X the spiking level?
 - i. If yes, the spike recovery is not evaluated.
 - ii. If no, a post digestion spike must be analyzed and the associated sample data must be qualified on the final report.

19.3.3. Post digestion spike analysis recovery, when required

- a. The associated sample data must be qualified on the final report.
- b. For USACE, analysis by MSA is required.

19.3.4. Serial dilution analysis

- a. Is the analyte concentration a factor of 50 above the instrumental detection limit after dilution?
 - i. If no, the serial dilution data can not be evaluated.
 - ii. If yes, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

20. Waste Management

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21. References

21.1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III)*; Method 6010B and Method 6010C.

21.2. USEPA *Code of Federal Regulations, 40, CH 1, PT 136*; Method 200.7; APX-B.

21.3. DOD Quality Systems Manual for Environmental Laboratories Version 4.2. (Based on NELAC Voted Revision June 5, 2003. 10/25/2010)

21.4. Standard Methods – 19th, 20th, 21st and 22nd Editions.

22. Tables, Diagrams, Flowcharts and Validation Data

Table 1 contains all applicable parameters with the MDL/DL, LOD, MRL/LOQ

Table 1A, contains a list of the wavelengths used for each analyte.

Table 1B, IEC listing indicating metals affected when a certain metal is present.

Table 1C, Instrument Software Flags

Table 2, QA/QC summary table.

Table 3, Technical Completeness / Accuracy Checklist

Table 4, Data Reviewers Checklist

Appendix I, Calibration Standard preparation

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Table 1 - MDL/DL, LOD, MRL/LOQ Limits

S/W	Method	Analyte	MDL/DL	LOD	MRL/LOQ	Units
Solid	6010B/C	Aluminum	10.0	20.0	40.0	mg/Kg
Solid	6010B/C	Antimony	1.00	1.60	2.00	mg/Kg
Solid	6010B/C	Arsenic	0.600	1.20	2.00	mg/Kg
Solid	6010B/C	Barium	1.00	4	8.00	mg/Kg
Solid	6010B/C	Beryllium	0.200	0.400	1.00	mg/Kg
Solid	6010B/C	Boron	2.00	4.00	8	mg/Kg
Solid	6010B/C	Cadmium	0.200	0.400	1	mg/Kg
Solid	6010B/C	Calcium	200	400	1000	mg/Kg
Solid	6010B/C	Chromium, total	0.400	0.800	2.00	mg/Kg
Solid	6010B/C	Cobalt	1.00	2.00	2.50	mg/Kg
Solid	6010B/C	Copper	0.800	1.60	2.00	mg/Kg
Solid	6010B/C	Iron	6.00	12.0	20.0	mg/Kg
Solid	6010B/C	Lead	0.300	0.600	1	mg/Kg
Solid	6010B/C	Magnesium	200	600	1000	mg/Kg
Solid	6010B/C	Manganese	0.600	1.20	3.00	mg/Kg
Solid	6010B/C	Molybdenum	1.00	2.00	4	mg/Kg
Solid	6010B/C	Nickel	0.600	1.20	2.00	mg/Kg
Solid	6010B/C	Potassium	200	600	1000	mg/Kg
Solid	6010B/C	Selenium	0.600	1.00	2	mg/Kg
Solid	6010B/C	Silver	0.200	0.400	2.00	mg/Kg
Solid	6010B/C	Sodium	200	600	1000	mg/Kg
Solid	6010B/C	Strontium	0.300	0.600	1.20	mg/Kg
Solid	6010B/C	Thallium	0.600	0.800	1.60	mg/Kg
Solid	6010B/C	Tin	2.00	10	40	mg/Kg
Solid	6010B/C	Titanium	1.00	2.00	4	mg/Kg
Solid	6010B/C	Vanadium	1.00	2.00	2.50	mg/Kg
Solid	6010B/C	Zinc	1.00	2.00	4.00	mg/Kg
Water	6010B/C	Aluminum	50.0	100	200	ug/L
Water	6010B/C	Antimony	5.00	8.00	10	ug/L
Water	6010B/C	Arsenic	3.00	6.00	10.0	ug/L
Water	6010B/C	Barium	5.00	10.0	40.0	ug/L
Water	6010B/C	Beryllium	1.00	2.00	5.00	ug/L
Water	6010B/C	Boron	10.0	20.0	40	ug/L
Water	6010B/C	Cadmium	1.00	2.00	5.00	ug/L
Water	6010B/C	Calcium	1000	2000	5000	ug/L
Water	6010B/C	Chromium, total	2.00	4.00	10.0	ug/L
Water	6010B/C	Cobalt	5.00	10.0	12.5	ug/L
Water	6010B/C	Copper	4.00	8.00	10.0	ug/L
Water	6010B/C	Iron	30.0	60.0	100	ug/L
Water	6010B/C	Lead	1.50	3.00	3.00	ug/L
Water	6010B/C	Magnesium	1000	3000	5000	ug/L
Water	6010B/C	Manganese	3.00	6.00	15.0	ug/L
Water	6010B/C	Molybdenum	5.00	10.0	20	ug/L
Water	6010B/C	Nickel	3.00	6.00	10.0	ug/L
Water	6010B/C	Potassium	1000	3000	5000	ug/L
Water	6010B/C	Selenium	3.00	5.00	10	ug/L
Water	6010B/C	Silver	1.00	2.00	10.0	ug/L

Table 1 – MDL/DL, LOD, MRL/LOQ Limits - Continued

S/W	Method	Analyte	MDL/DL	LOD	MRL/LOQ	Units
Water	6010B/C	Sodium	1000	3000	5000	ug/L
Water	6010B/C	Strontium	1.50	3.00	6.00	ug/L
Water	6010B/C	Thallium	3.00	4.00	8.00	ug/L
Water	6010B/C	Tin	10.0	20.0	30.0	ug/L
Water	6010B/C	Titanium	5.00	10.0	20	ug/L
Water	6010B/C	Vanadium	5.00	10.0	12.5	ug/L
Water	6010B/C	Zinc	5.00	10.0	20.0	ug/L
Solid	6010B/C low	Aluminum	2.5	5	10	mg/Kg
Solid	6010B/C low	Antimony	0.25	0.4	0.50	mg/Kg
Solid	6010B/C low	Arsenic	0.15	0.3	0.5	mg/Kg
Solid	6010B/C low	Barium	0.25	0.5	2	mg/Kg
Solid	6010B/C low	Beryllium	0.05	0.1	0.25	mg/Kg
Solid	6010B/C low	Boron	0.5	1	2	mg/Kg
Solid	6010B/C low	Cadmium	0.05	0.1	0.25	mg/Kg
Solid	6010B/C low	Calcium	50	100	250	mg/Kg
Solid	6010B/C low	Chromium, total	0.1	0.2	0.5	mg/Kg
Solid	6010B/C low	Cobalt	0.25	0.5	0.625	mg/Kg
Solid	6010B/C low	Copper	0.2	0.4	0.5	mg/Kg
Solid	6010B/C low	Iron	1.5	3	7.5	mg/Kg
Solid	6010B/C low	Lead	0.075	0.15	0.25	mg/Kg
Solid	6010B/C low	Magnesium	50	150	250	mg/Kg
Solid	6010B/C low	Manganese	0.15	0.3	0.75	mg/Kg
Solid	6010B/C low	Molybdenum	0.25	0.5	1	mg/Kg
Solid	6010B/C low	Nickel	0.15	0.3	0.5	mg/Kg
Solid	6010B/C low	Potassium	50	150	250	mg/Kg
Solid	6010B/C low	Selenium	0.15	0.25	0.5	mg/Kg
Solid	6010B/C low	Silver	0.05	0.1	0.5	mg/Kg
Solid	6010B/C low	Sodium	50	150	250	mg/Kg
Solid	6010B/C low	Strontium	0.075	0.15	0.3	mg/Kg
Solid	6010B/C low	Thallium	0.15	0.2	0.4	mg/Kg
Solid	6010B/C low	Tin	0.5	2.5	10	mg/Kg
Solid	6010B/C low	Titanium	0.25	0.5	1	mg/Kg
Solid	6010B/C low	Vanadium	0.25	0.5	0.625	mg/Kg
Solid	6010B/C low	Zinc	0.25	0.5	1	mg/Kg
Water	6010B/C low	Aluminum	12.5	25	50	ug/L
Water	6010B/C low	Antimony	1.25	2	2.5	ug/L
Water	6010B/C low	Arsenic	0.75	1.5	2.5	ug/L
Water	6010B/C low	Barium	1.25	2.5	10	ug/L
Water	6010B/C low	Beryllium	0.25	0.5	1.25	ug/L
Water	6010B/C low	Boron	2.5	5	10	ug/L
Water	6010B/C low	Cadmium	0.25	0.5	1.25	ug/L
Water	6010B/C low	Calcium	250	500	1250	ug/L
Water	6010B/C low	Chromium, total	0.5	1	2.5	ug/L
Water	6010B/C low	Cobalt	1.25	2.5	3.125	ug/L
Water	6010B/C low	Copper	1	2	2.5	ug/L
Water	6010B/C low	Iron	7.5	15	25	ug/L

Table 1 – MDL/DL, LOD, MRL/LOQ Limits - Continued

S/W	Method	Analyte	MDL/DL	LOD	MRL/LOQ	Units
Water	6010B/C low	Lead	0.375	0.75	0.75	ug/L
Water	6010B/C low	Magnesium	250	750	1250	ug/L
Water	6010B/C low	Manganese	0.75	1.5	3.75	ug/L
Water	6010B/C low	Molybdenum	1.25	2.5	5.0	ug/L
Water	6010B/C low	Nickel	0.75	1.5	2.5	ug/L
Water	6010B/C low	Potassium	250	750	1250	ug/L
Water	6010B/C low	Selenium	0.75	1.25	2.5	ug/L
Water	6010B/C low	Silver	0.25	0.5	2.5	ug/L
Water	6010B/C low	Sodium	250	750	1250	ug/L
Water	6010B/C low	Strontium	0.375	0.75	1.5	ug/L
Water	6010B/C low	Thallium	0.75	1	2.0	ug/L
Water	6010B/C low	Tin	2.5	5	7.5	ug/L
Water	6010B/C low	Titanium	1.25	2.5	5	ug/L
Water	6010B/C low	Vanadium	1.25	2.5	3.125	ug/L
Water	6010B/C low	Zinc	1.25	2.5	5.0	ug/L
TCLP	SW6010B/C_1311	Arsenic	0.03	0.06	0.1	mg/L
TCLP	SW6010B/C_1311	Barium	0.05	0.1	0.4	mg/L
TCLP	SW6010B/C_1311	Cadmium	0.01	0.02	0.05	mg/L
TCLP	SW6010B/C_1311	Chromium	0.02	0.04	0.1	mg/L
TCLP	SW6010B/C_1311	Lead	0.015	0.03	0.03	mg/L
TCLP	SW6010B/C_1311	Selenium	0.030	0.05	0.1	mg/L
TCLP	SW6010B/C_1311	Silver	0.01	0.02	0.1	mg/L

TABLE 1A

METAL	WAVELENGTH
Aluminum	396.1
Antimony	206.8
Arsenic	189.0
Barium	233.5
Beryllium	313.0
Boron	249.7
Cadmium	214.4
Calcium	317.9
Chromium	267.7
Cobalt	228.6
Copper	324.7
Iron	261.1
Lead	220.3
Magnesium	279.0
Manganese	257.6
Molybdenum	202.0
Nickel	231.6
Potassium	766.4
Selenium	196.0
Silver	328.0
Sodium	589.5
Strontium	421.5
Thallium	190.8
Tin	189.9
Titanium	334.9
Vanadium	292.4
Zinc	206.2

Table 1B – IEC listing indicating metals affected when a certain metal is present.

6500 Duo IECs
Metals Affected

Ag	_____
Al	_____ As, Pb, Sb, Se, Ti, Zn
As	_____ Cd
B	_____
Ba	_____
Be	_____ Zn
Cu	_____ Al, Pb, Ti, Zn, Ni, Se, Sr
Cd	_____ Cr
Co	_____ Cd, Pb, Ti
Cr	_____ As, Ba, Fe, Ni, Sb, Ti, Ti, V, Zn
Cu	_____ Pb, Zn
Fe	_____ Ag, Al, As, B, Ba, Cd, Co, Cr, Cu, Mg, Mn, Mo, Ni, Pb, Sb, Se, Ti, V, Zn
K	_____
Mg	_____ Cd, Cr, Pb, Ti, Zn
Mn	_____ Ag, Cr, Fe, Pb, Se, Ti, V
Mo	_____ Ag, Al, As, Be, Cd, Co, Cu, Mg, Ni, Pb, Sb, Se, Ti, V, Zn
Na	_____
Ni	_____ Cd, Co, Pb, Se
Pb	_____
Sb	_____ Ni, Zn
Se	_____ Zn
Sn	_____
Ti	_____ As, Be, Co, Cu, Pb, Sb, Se, Ti, V
Tl	_____ Ni
V	_____ Ag, Ba, Be, Cd, Cr, Cu, Mo, Pb, Sb, Se, Ti, Zn
Zn	_____ Ni, As

Table 1C – Instrument Software Flags

ITEVA Analysis Flags

The following errors may be encountered during analysis:

Error Flag Reason

F	The check has failed the limits set in the Check Table.
W	The warning limit set in the Check Table has been reached/exceeded.
>	The result is above the print limits set for the method.
<	The result is below the print limits set for the method.
? value	There is a calculation error associated with the result (could be due to an invalid calibration, IEC etc.).
C	The concentration is beyond the calibrated range (past inflection point) .
c	The concentration yields negative value (below blank standard) .
z	The data does not have a valid calibration associated with it (this will happen if samples are run before a method is calibrated, unless the Output Mode is set to an option other than Concentration in Method/ Report Preferences/Output Mode).
K	There is an error on an interferent line. This error is also reported if the IEC threshold has been exceeded. See Method/Element/General/IEC/Threshold .
I	Interferent (overlapping Subarrays) - data can be used. This error is due to choosing two or more analytical lines that overlap with each other spectrally. Ideally, an alternate line should be chosen see Method/View and refer to the Interference Tables, or you can choose to apply an IEC correction to overcome the interference. See Method/Elements/IEC .
I	Interferent (overlapping subarrays) - data cannot be used as the peaks cannot be resolved.
D	The analyte peak is off the CID detector.
^	The peak is saturated (data not returned).
P	Plasma error, the plasma was extinguished during analysis.
*	Global failure - NO DATA ACQUIRED. May be due to a memory error due to too many data points.
S	Error on internal standard line.
Z	Internal standard error, normally due to an invalid calibration or incorrectly set up internal standard reference. This flag is also displayed if an analysis is run without a valid calibration.
N	The wavelength was de-selected before exposure, no data returned.
R	The wavelength failed intelligent rinse test (i.e. the maximum no. of attempts was exceeded).

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Interference Check	<ul style="list-style-type: none"> once per calibration 	<ul style="list-style-type: none"> IFA less than LOD if not verified contamination of standard. IFB must be within $\pm 20\%$. 	<ul style="list-style-type: none"> Check IEC corrections for metals in the IFA.
Calibration Curve	<ul style="list-style-type: none"> Prior to analyzing any samples A minimum of a blank and 3-points for linear fits client specific requirement or a blank and high standard. 	<ul style="list-style-type: none"> Linear calibration Corr. of 0.998 Must follow curve processing requirements from SOP QS08 	<ul style="list-style-type: none"> Re-evaluate curve mix and makeup Re-run curve Check instrument for maintenance needs Re-prepare the curve standards <p>Samples cannot be analyzed until there is a passing calibration</p>
ICB/CCB	<ul style="list-style-type: none"> At the beginning of every sequence For every 10-field samples 	Must meet the $\leq \pm \text{LOD}$ for DOD or $< 2 \times \text{MDL/DL}$	Re-run
ICV	Alternate source standard to be analyzed after every calibration curve	<ul style="list-style-type: none"> Must be in the range 90 to 110% for 6010B/6010C/DoD, or 95 to 105% for 200.7. 	<ul style="list-style-type: none"> Re-analyze an ICV from a different source Re-prepare and re-analyze the ICV Re-calibrate and verify standard preps and sources
LLICV	Low standard at the LOQ/RL level run against the curve.	<ul style="list-style-type: none"> Within 20% DoD, 30% for 6010C to report at this LOQ/RL. 	<ul style="list-style-type: none"> Re-analyze an ICV from a different source Re-prepare and re-analyze the ICV Re-calibrate and verify standard preps and sources Raise LOQ/RL to next passing level.
CCV	<ul style="list-style-type: none"> At the beginning of every sequence For every 10-field samples 	<ul style="list-style-type: none"> Must be in the range 90 to 110% 	<ul style="list-style-type: none"> Samples must be reanalyzed if possible, if not samples are qualified with a "X" (high) or "Y" (low).
Closing CCV	<ul style="list-style-type: none"> At the end of every sequence 	<ul style="list-style-type: none"> Must be in the range 90 to 110% 	<ul style="list-style-type: none"> Samples must be reanalyzed if possible, if not samples are qualified with a "X" (high) or "Y" (low).
Closing LLCCV	<ul style="list-style-type: none"> At the end of every 6010C sequence 	<ul style="list-style-type: none"> Should be in the range 70 to 130% for 6010C 	<ul style="list-style-type: none"> Samples must be reanalyzed if possible, if not samples are qualified with a "X" (high) or "Y" (low).

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
BLK	One per prep batch	<ul style="list-style-type: none"> Must be less than $\frac{1}{2} \pm RL$ (project reporting limit) for DoD, and $< \pm 2 \times MDL$ for all others 	<ul style="list-style-type: none"> Re-analysis to confirm the positive value Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action Re-prep of samples associated with the MB NCR will be required for data reported Final Report data qualification will be required
BS	One per prep batch	Must be in the range of 80 to 120% for 6010B/6010C/DOD; or 85 to 115% for 200.7.	<ul style="list-style-type: none"> Rerun to confirm problem. All samples associated with the LCS must be re-digested, reanalyzed if possible. NCR will be required for data reported If samples cannot be re-digested or re-analyzed Final Report data qualification will be required
MS	One per prep batch	Must be in the range of 80 to 120%	Final Report data qualification will be required
MSD	One per prep batch	Must be in the range of 80 to 120%	Final Report data qualification will be required
Sample Duplicate	One per prep batch	20%	Qualify samples
Dilution test (Only applicable for samples with concentrations $> 50 \times LOQ/MRL$)	One per prep batch	Five-fold dilution must agree within $\pm 10\%$ of the original measurement.	Perform post-digestion spike (PDS) addition;
Post Digestion Spike	One per prep batch (When dilution test fails or analyte concentration in all samples $< 50 \times LOD$.)	$\pm 25\%$ for DOD/6010B, $\pm 20\%$ 6010C	For the specific analyte(s) in the parent sample, apply J-qualify if acceptance criteria are not met.
DOC Study	<ul style="list-style-type: none"> Initially per analyst prior to reporting data Annually Follow specific guidelines from section 16 for the preparation and analysis of DOC samples 	<ul style="list-style-type: none"> Must meet the criteria of the BS for average accuracy 	<ul style="list-style-type: none"> Re-prep and / or Re-analysis
MDL Study	Annually		
LOD Verification	Every quarter	Must be spiked at the LOD concentration and meet requirements for detection	

Table 2 - Method Quality Control Requirements Summary			
QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
LOQ Verification	Every quarter	Must be spiked at 1-2 times the LOQ concentration and meet requirements for LCS recovery.	
Linear Dynamic Range Study (LDR)	Every six months	Within $\pm 10\%$ of true value.	

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were dilution factors applied correctly
5. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
6. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
7. Were proper data qualifiers applied to the data in LIMS
8. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

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Table 4

ANALYST DATA REVIEW CHECKLIST Sample Number(s):				
Batch Number(s):		Sequence ID:		
Method: 6010B or 6010C (ICP)				
QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within holding times?				
2. Was initial calibration curve QC criteria met?				
3. Was all continuing calibration criteria in control?				
4. Did any sample exceed the highest calibration standard?				
(If yes, were appropriate dilutions made to generate samples concentration within calibration range?)				
5. Did BS (blank spike) meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation (Method) Blank (BLK) below the necessary limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was hot plate temperature monitored/documented and did you apply the thermometer correction factor?				
10. Sample preparation information is correct and complete.				
11. Analytical results are correct and complete.				
12. The appropriate SOP's have been followed.				
14. "Raw data" including all manual integration's have been correctly interpreted.				
15. "Special" sample preparation and analytical requirements have been met and documented.				
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, non-conformance reports are complete.				
17. Data has been uploaded to the LIMS with correct analyst and analysis factors included.				
18. Calculation is shown on the raw data for sample _____ to validate final concentration in LIMS.				

Comments on any "No" response:

Analyst: _____ Date: _____

APPENDIX I

Preparation Method for Calibration Standards

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Analytical Standard Record
Empirical Laboratories, LLC
12G0052

Description:	SEQ-CAL2	Expires:	07/17/2012
Standard Type:	Calibration Stan	Prepared:	07/03/2012
Solvent:	2% HNO3 5% HCl	Prepared By:	Roger Burr
Final Volume (mL):	100	Department:	METALS
Vials:	1	Last Edit:	07/18/2012 08:46 by RGB

Analyte	CAS Number	Concentration	Units
Magnesium	7439-95-4	5.1	mg/L
Antimony	7440-36-0	0.1	mg/L
Arsenic	7440-38-2	0.1	mg/L
Barium	7440-39-3	0.05	mg/L
Beryllium	7440-41-7	0.1	mg/L
Boron	7440-42-8	0.05	mg/L
Cadmium	7440-43-9	0.1	mg/L
Calcium	7440-70-2	1.1	mg/L
Chromium	7440-47-3	0.1	mg/L
Cobalt	7440-48-4	0.1	mg/L
Copper	7440-50-8	0.1	mg/L
Iron	7439-89-6	5.1	mg/L
Aluminum	7429-90-5	5	mg/L
Lithium	7439-93-2	0.1	mg/L
Zinc	7440-66-6	0.1	mg/L
Manganese	7439-96-5	0.1	mg/L
Molybdenum	7439-98-7	0.1	mg/L
Nickel	7440-02-0	0.1	mg/L
Potassium	7440-09-7	1	mg/L
Selenium	7782-49-2	0.1	mg/L
Silver	7440-22-4	0.02	mg/L
Sodium	7440-23-5	1	mg/L
Strontium	7440-24-6	0.1	mg/L
Thallium	7440-28-0	0.1	mg/L
Tin	7440-31-5	0.05	mg/L
Titanium	7440-32-6	0.1	mg/L
Vanadium	7440-62-2	0.1	mg/L
Lead	7439-92-1	0.1	mg/L

Analytical Standard Record
Empirical Laboratories, LLC
12G0052

Parent Standards used in this standard:					
Standard	Description	Prepared	Prepared By	Expires	Last Edit (m/s)
12D0517	SEQ-CAL7	04/12/2012	Roger Burr	07/31/2012	07/18/2012 08:46 by RGB
12D0447	Ag Ba CAL STOCK	04/17/2012	Roger Burr	07/17/2012	04/17/2012 08:07 by RGB
12G0045	SEQ-CAL6	07/03/2012	Roger Burr	10/03/2012	07/03/2012 10:16 by RGB
12G0046	SEQ-CAL5	07/03/2012	Roger Burr	10/03/2012	07/03/2012 10:19 by RGB

Analytical Standard Record
Empirical Laboratories, LLC
12F0296

Description:	SEQ-CAL3	Expires:	09/19/2012
Standard Type:	Calibration Stan	Prepared:	06/19/2012
Solvent:	2% HNO3 5% HCl	Prepared By:	Roger Burr
Final Volume (mils):	100	Department:	METALS
Vials:	1	Last Edit:	06/19/2012 08:30 by RGB

Analyte	CAS Number	Concentration	Units
Manganese	7439-96-5	1	ppm
Antimony	7440-36-0	1	ppm
Arsenic	7440-38-2	1	ppm
Barium	7440-39-3	1	ppm
Beryllium	7440-41-7	1	ppm
Boron	7440-42-8	1	ppm
Cadmium	7440-43-9	1	ppm
Calcium	7440-70-2	50	ppm
Chromium	7440-47-3	1	ppm
Cobalt	7440-48-4	1	ppm
Copper	7440-50-8	1	ppm
Iron	7439-89-6	10	ppm
Lead	7439-92-1	1	ppm
Aluminum	7429-90-5	10	ppm
Magnesium	7439-95-4	50	ppm
Zinc	7440-66-6	1	ppm
Molybdenum	7439-98-7	1	ppm
Nickel	7440-02-0	1	ppm
Potassium	7440-09-7	10	ppm
Selenium	7782-49-2	1	ppm
Silicon	7440-21-3	1	ppm
Silver	7440-22-4	0.5	ppm
Sodium	7440-23-5	50	ppm
Strontium	7440-24-6	1	ppm
Thallium	7440-28-0	1	ppm
Tin	7440-31-5	1	ppm
Titanium	7440-32-6	1	ppm
Vanadium	7440-62-2	1	ppm
Lithium	7439-93-2	1	ppm

Analytical Standard Record
Empirical Laboratories, LLC
12F0296

Parent Standards used in this standard:						
Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mils)
110590	Sodium	09/22/2011	Roger Burr	03/15/2013	09/22/2011 17:03 by RGB	0.49
11J0586	QC21	10/21/2011	Roger Burr	10/30/2012	10/21/2011 09:29 by RGB	1
11K0001	QCS7M	11/01/2011	Roger Burr	10/27/2012	11/01/2011 07:20 by RGB	1
11K0003	Calcium	11/01/2011	Roger Burr	04/27/2013	11/01/2011 07:24 by RGB	0.49
12B0334	Aluminum	02/13/2012	Roger Burr	02/15/2013	02/13/2012 15:12 by RGB	0.09
12B0336	Iron	02/13/2012	Roger Burr	02/15/2013	02/13/2012 15:13 by RGB	0.09
12B0592	Magnesium	02/20/2012	Roger Burr	08/16/2013	02/20/2012 15:46 by RGB	0.49
12F0047	SN109	06/05/2012	Roger Burr	09/05/2012	06/05/2012 09:14 by RGB	1

Analytical Standard Record
Empirical Laboratories, LLC
12F0288

Description:	SEQ-CAL4	Expires:	09/19/2012
Standard Type:	Calibration Stan	Prepared:	06/19/2012
Solvent:	2% HNO3 5% HCl	Prepared By:	Roger Burr
Final Volume (mls):	200	Department:	METALS
Vials:	1	Last Edit:	06/19/2012 07:56 by RGB

Analyte	CAS Number	Concentration	Units
Silver	7440-22-4	2	ppm
Manganese	7439-96-5	10	ppm
Barium	7440-39-3	5	ppm

Parent Standards used in this standard:						
Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
11G0771	SILVER	07/29/2011	Roger Burr	01/22/2013	07/29/2011 14:33 by RGB	0.4
11K0002	BARIUM	11/01/2011	Roger Burr	04/27/2013	11/01/2011 07:22 by RGB	0.1
12B0589	MANGANESE	02/20/2012	Roger Burr	08/16/2013	02/20/2012 15:41 by RGB	2

Analytical Standard Record
Empirical Laboratories, LLC
12G0046

Description:	SEQ-CAL5	Expires:	10/03/2012
Standard Type:	Calibration Stan	Prepared:	07/03/2012
Solvent:	2% HNO3 5% HCl	Prepared By:	Roger Burr
Final Volume (mls):	500	Department:	METALS
Vials:	1	Last Edit:	07/03/2012 10:19 by RGB

Analyte	CAS Number	Concentration	Units
Magnesium	7439-95-4	500	ppm
Iron	7439-89-6	500	ppm
Aluminum	7429-90-5	500	ppm

Parent Standards used in this standard:						
Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
11G0766	ALUMINUM	07/29/2011	Roger Burr	01/22/2013	07/29/2011 14:29 by RGB	25
11L0203	IRON	12/07/2011	Roger Burr	05/29/2013	12/07/2011 10:07 by RGB	25
12B0592	Magnesium	02/20/2012	Roger Burr	08/16/2013	02/20/2012 15:46 by RGB	25

**Analytical Standard Record
Empirical Laboratories, LLC
12G0045**

Description:	SEQ-CAL6	Expires:	10/03/2012
Standard Type:	Calibration Stan	Prepared:	07/03/2012
Solvent:	2% HNO3 5% HCl	Prepared By:	Roger Burr
Final Volume (mls):	500	Department:	METALS
Vials:	1	Last Edit:	07/03/2012 10:16 by RGB

Analyte	CAS Number	Concentration	Units
Magnesium	7439-95-4	10	mg/L
Arsenic	7440-38-2	10	mg/L
Beryllium	7440-41-7	10	mg/L
Boron	7440-42-8	5	mg/L
Cadmium	7440-43-9	10	mg/L
Calcium	7440-70-2	10	mg/L
Chromium	7440-47-3	10	mg/L
Cobalt	7440-48-4	10	mg/L
Copper	7440-50-8	10	mg/L
Iron	7439-89-6	10	mg/L
Antimony	7440-36-0	10	mg/L
Lithium	7439-93-2	10	mg/L
Zinc	7440-66-6	10	mg/L
Manganese	7439-96-5	10	mg/L
Molybdenum	7439-98-7	10	mg/L
Nickel	7440-02-0	10	mg/L
Selenium	7782-49-2	10	mg/L
Strontium	7440-24-6	10	mg/L
Thallium	7440-28-0	10	mg/L
Tin	7440-31-5	5	mg/L
Titanium	7440-32-6	10	mg/L
Vanadium	7440-62-2	10	mg/L
Lead	7439-92-1	10	mg/L

**Analytical Standard Record
Empirical Laboratories, LLC
12G0045**

Parent Standards used in this standard:						
Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
11F0749	TIN	06/24/2011	Roger Burr	12/20/2012	06/24/2011 16:05 by RGB	0.25
11J0586	QC21	10/21/2011	Roger Burr	10/30/2012	10/21/2011 09:29 by RGB	50
12B0588	Boron	02/20/2012	Roger Burr	08/16/2013	02/20/2012 15:40 by RGB	2.5

**Analytical Standard Record
Empirical Laboratories, LLC
12D0317**

Description:	SEQ-CAL7	Expires:	07/31/2012
Standard Type:	Calibration Stan	Prepared:	04/12/2012
Solvent:	2% HNO3 5% HCl	Prepared By:	Roger Burr
Final Volume (mls):	500	Department:	METALS
Vials:	1	Last Edit:	07/18/2012 08:46 by RGB

Analyte	CAS Number	Concentration	Units
Sodium	7440-23-5	100	ppm
Potassium	7440-09-7	100	ppm
Calcium	7440-70-2	100	ppm

Parent Standards used in this standard:						
Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
11D0103	Potassium	02/04/2011	Roger Burr	07/31/2012	02/04/2011 14:07 by RGB	5
11F0751	Sodium	06/24/2011	Roger Burr	12/20/2012	06/24/2011 16:30 by RGB	5
12B0591	Calcium	02/20/2012	Roger Burr	08/16/2013	02/20/2012 15:45 by RGB	5

Analytical Standard Record
Empirical Laboratories, LLC
12E0004

Description:	SEQ-CAL8	Expires:	08/01/2012
Standard Type:	Calibration Stan	Prepared:	05/01/2012
Solvent:	2% HNO3 5% HCL	Prepared By:	Roger Burr
Final Volume (mls):	500	Department:	METALS
Vials:	1	Last Edit:	05/01/2012 08:30 by RGB

Analyte	CAS Number	Concentration	Units
Sodium	7440-23-5	500	ppm
Magnesium	7439-95-4	100	ppm
Calcium	7440-70-2	500	ppm

Parent Standards used in this standard:					
Standard	Description	Prepared	Prepared By	Expires	Last Edit (mls)
11K0603	Calcium	11/01/2011	Roger Burr	04/27/2013	11/01/2011 07:24 by RGB 25
12D0590	Sodium	02/20/2012	Roger Burr	08/16/2013	02/20/2012 15:43 by RGB 25
12D0592	Magnesium	02/20/2012	Roger Burr	08/16/2013	02/20/2012 15:46 by RGB 5

Analytical Standard Record
Empirical Laboratories, LLC
12G0359

Description:	ICV METALS	Expires:	07/19/2012
Standard Type:	Calibration Stan	Prepared:	07/18/2012
Solvent:	2% HNO3 5% HCL	Prepared By:	Roger Burr
Final Volume (mls):	100	Department:	METALS
Vials:	1	Last Edit:	07/18/2012 08:46 by RGB

Analyte	CAS Number	Concentration	Units
Manganese	7439-96-5	1	ppm
Antimony	7440-36-0	1	ppm
Arsenic	7440-38-2	1	ppm
Barium	7440-39-3	1	ppm
Beryllium	7440-41-7	1	ppm
Boron	7440-42-8	1	ppm
Cadmium	7440-43-9	1	ppm
Calcium	7440-70-2	50	ppm
Chromium	7440-47-3	1	ppm
Cobalt	7440-48-4	1	ppm
Copper	7440-50-8	1	ppm
Iron	7439-89-6	10	ppm
Lead	7439-92-1	1	ppm
Aluminum	7429-90-5	10	ppm
Magnesium	7439-95-4	50	ppm
Zinc	7440-66-6	1	ppm
Molybdenum	7439-98-7	1	ppm
Nickel	7440-02-0	1	ppm
Phosphorus, Total (as P)	7723-14-0	1	ppm
Potassium	7440-09-7	10	ppm
Selenium	7782-49-2	1	ppm
Silicon	7440-21-3	5	ppm
Silver	7440-22-4	0.5	ppm
Sodium	7440-23-5	50	ppm
Strontium	7440-24-6	1	ppm
Thallium	7440-28-0	1	ppm
Tin	7440-31-5	1	ppm
Titanium	7440-32-6	1	ppm
Vanadium	7440-62-2	1	ppm
Lithium	7439-91-2	1	ppm

Analytical Standard Record
Empirical Laboratories, LLC
12G0359

Parent Standards used in this standard:						
Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
11D0726	QC23	04/29/2011	Roger Burr	07/31/2012	04/29/2011 15:34 by RGB	1
11B0587	Magnesium	09/22/2011	Roger Burr	03/15/2013	09/22/2011 16:59 by RGB	0.49
11B0590	Sodium	09/22/2011	Roger Burr	03/15/2013	09/22/2011 17:03 by RGB	0.49
11K0003	Calcium	11/01/2011	Roger Burr	04/27/2013	11/01/2011 07:24 by RGB	0.49
12B0334	Aluminum	02/13/2012	Roger Burr	02/15/2013	02/13/2012 15:12 by RGB	0.09
12B0336	Iron	02/13/2012	Roger Burr	02/15/2013	02/13/2012 15:13 by RGB	0.09
12B0687	QC57A	02/23/2012	Roger Burr	02/28/2013	02/23/2012 16:00 by RGB	1

Analytical Standard Record
Empirical Laboratories, LLC
12G0360

Description:	CCV METALS	Expires:	07/19/2012
Standard Type:	Calibration Stan	Prepared:	07/18/2012
Solvent:	2% HNO3 5% HCl	Prepared By:	Roger Burr
Final Volume (ml):	100	Department:	METALS
Vials:	1	Last Edit:	07/18/2012 08:47 by RGB

Analyte	CAS Number	Concentration	Units
Manganese	7439-96-5	1	ppm
Antimony	7440-36-0	1	ppm
Arsenic	7440-38-2	1	ppm
Barium	7440-39-3	1	ppm
Beryllium	7440-41-7	1	ppm
Boron	7440-42-8	1	ppm
Cadmium	7440-43-9	1	ppm
Calcium	7440-70-2	50	ppm
Chromium	7440-47-3	1	ppm
Cobalt	7440-48-4	1	ppm
Copper	7440-50-8	1	ppm
Iron	7439-89-6	10	ppm
Lead	7439-92-1	1	ppm
Aluminum	7429-90-5	10	ppm
Magnesium	7439-95-4	50	ppm
Zinc	7440-66-6	1	ppm
Molybdenum	7439-98-7	1	ppm
Nickel	7440-02-0	1	ppm
Potassium	7440-09-7	10	ppm
Selenium	7782-49-2	1	ppm
Silicon	7440-21-3	1	ppm
Silver	7440-22-4	0.5	ppm
Sodium	7440-23-5	50	ppm
Strontium	7440-24-6	1	ppm
Thallium	7440-28-0	1	ppm
Tin	7440-31-5	1	ppm
Titanium	7440-32-6	1	ppm
Vanadium	7440-62-2	1	ppm
Lithium	7439-93-2	1	ppm

Analytical Standard Record
Empirical Laboratories, LLC
12G0360

Parent Standards used in this standard:						
Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
11B0590	Sodium	09/22/2011	Roger Burr	03/15/2013	09/22/2011 17:03 by RGB	0.49
11J0586	QC21	10/21/2011	Roger Burr	10/30/2012	10/21/2011 09:29 by RGB	1
11K0001	QC57M	11/01/2011	Roger Burr	10/27/2012	11/01/2011 07:20 by RGB	1
11K0003	Calcium	11/01/2011	Roger Burr	04/27/2013	11/01/2011 07:24 by RGB	0.49
12B0334	Aluminum	02/13/2012	Roger Burr	02/15/2013	02/13/2012 15:12 by RGB	0.09
12B0336	Iron	02/13/2012	Roger Burr	02/15/2013	02/13/2012 15:13 by RGB	0.09
12B0592	Magnesium	02/20/2012	Roger Burr	08/16/2013	02/20/2012 15:46 by RGB	0.49
12F0047	SN100	06/05/2012	Roger Burr	06/05/2012	06/05/2012 09:14 by RGB	1

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

INORGANICS: SOP 154

REVISION #: 09

EFFECTIVE DATE: 20130410

TOTAL ALKALINITY, CARBONATE, BICARBONATE

METHOD: SM 2320 B-2011

APPROVALS:

Lab Director: _____

Date: 04/10/2013

Data Quality Manager: _____

Date: 04/10/2013

Section Supervisor: _____

Date: 04/10/2013

Changes Summary

Revision 09, 04/10/13

- Removed references to EPA method 310.1 and updated standard methods reference format
- Section 7.3 updated to reflect all known interference possibilities.
- Section 9.5 updated to add Temperature probe.
- Sections 10.3 and 10.4 updated to add mg CaCO₃ equivalents.
- Reference to duplicate removed and replaced with MS/MSD references in section 12.3.
- Spreadsheet directory updated in section 12.4.
- Calibration section 14.5 updated.
- Wording of section 14.6 updated.

Revision 08, 09/16/11

- The SOP is an update from Revision 07 dated 09/21/2010
- Revised pH probe to pH and temperature probe in Equipment and Supplies section.

Revision 07, 09/21/10

- The SOP is an update from Revision 06 dated 09/07/2010
- Incorrect reference to GC/FID in section 1.1 was updated to titration.
- References to preparation blanks as PB were updated to BLK and references to laboratory control samples were updated to blank spikes/blank spike duplicates as BS/BSD.
- Magnetic stirrer and stirbars were added to equipment list.
- Units were updated from mg/L to mg CaCO₃/L.
- Table 2 criteria were updated for method specifics.

Revision 06, 09/07/10

- The SOP is an update from Revision 05 dated 05/27/2009
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
3. Detection Limit
4. Scope of Application, Including components to be Analyzed
5. Summary of the Test Method
6. Definitions
7. Interferences
8. Safety
9. Equipment & Supplies
10. Reagents and Standards
11. Sample Collection, Preservation, Shipment, and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Data Analysis and Calculations
16. Method Performance
17. Pollution Prevention
18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Contingencies for Handling out-of-control or unacceptable data
20. Waste Management
21. References
22. Tables, Diagrams, Flowcharts and Validation Data

1.0 Identification of the Test Method

- 1.1 The titration method is used to analyze TOTAL ALKALINITY, CARBONATE, BICARBONATE utilizing Standard Methods 2320B

2.0 Applicable Matrix or Matrices

2.1 This method is applicable to drinking, surface, and saline waters, and domestic and industrial wastes. Soils are leached 10 grams to 100 mLs and the analysis performed on the leachate.

2.2 The method is suitable for all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater than 10 mL.

3.0 Detection Limit

- 3.1 The detection limit is 1.0 mg/L CaCO_3 .

4.0 Scope of Application, Including Components to Be Analyzed

- 4.1 Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

5.0 Summary of the Test Method

- 5.1 When the sample is being analyzed for phenolphthalein alkalinity, carbonate, bicarbonate and total alkalinity, an unaltered sample is titrated to an electrometrically-determined endpoint of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way. The sample is then titrated to a pH exactly 0.3 pH units lower and the calculation for 2320B (in calculation section of this SOP) is used to calculate the samples for phenolphthalein alkalinity, total alkalinity, carbonate and bicarbonate results. See 14.14.3 for samples with pH greater than 8.3.

6.0 Definitions

- 6.1 Laboratory Quality System SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7.0 Interferences

- 7.1 Substances, such as salts or weak organic and inorganic acids present in large amounts, may cause interference in the electrometric pH measurements.
- 7.2 For samples having high concentrations of mineral acids, such as mine wastes and associated receiving waters, titrate to an electrometric endpoint of pH 3.9, using the procedure in *Annual Book of ASTM Standards*, Part 31, "Water," p. 115, D-1067, Method D (1976).
- 7.3 Soaps, oily matter, suspended solids, or precipitates may coat the pH electrode, and cause a sluggish response. Allow additional time between titrant additions to let electrode come to equilibrium or clean the electrode occasionally. Do not filter, dilute, concentrate, or alter sample.

8.0 Safety

- 8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.
- 8.2 Care should be used in handling all samples.
- 8.3 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
- 8.4 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.
- 8.5 Your laboratory manager and/or Safety Officer is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A formal safety plan is also available. Use proper personal protection equipment, PPE, such as safety glasses, gloves and laboratory coats should be worn when handling samples and chemicals.

9.0 Equipment & Supplies

- 9.1 pH meter that uses a glass electrode and can be read to 0.01 pH units. The analyst will note on the data which pH meter (either the Corning 240 or Orion 420A) is used.
- 9.2 50 mL disposable beakers with wide enough mouths to allow room for burette tip and pH and temperature probe.
- 9.3 10 mL Class A microburette.
- 9.4 Magnetic Stirrer and tiny stir bars.
- 9.5 Temperature probe.

10.0 Reagents and Standards

- 10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

ACS reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

Certified stock standards are purchased from Ultra Scientific, NSI, Ricca and other vendors depending on their availability. The date they are received is noted on the label or container they are received in, on the certificate of analysis (COA) and in the LIMS system. They are given a sequential number in the LIMS system which is also noted on the container and the COA which is

then scanned and stored in the LIMS system. The date the standards are opened is recorded in the LIMS system. All stock standards are stored at 4°C.

- 10.2 Sodium carbonate solution, approximately 0.05N: Place 2.5 ± 0.2 g (to nearest mg) Na_2CO_3 (dried at 250°C for 4 hours and cooled in desiccator) into a 1-liter, Class A, volumetric flask and dilute to the mark. The Na_2CO_3 solution must be disposed of after one week.
- 10.3 Standard Acid (sulfuric or hydrochloric), 0.1N (high titrant): Dilute 3.0 mL concentrated H_2SO_4 or 8.3 mL concentrated HCl to 1 liter with distilled water. Dilute 40 mL of 0.05N Na_2CO_3 solution to 100 mL with deionized water and titrate potentiometrically with the Standard Acid to a pH of about 5. Lift electrode and rinse into beaker. Boil solution gently for 3 to 5 minutes under a watch glass cover. Cool to room temperature. Rinse cover glass into beaker. Continue titration to the pH Inflection point (3 units lower). This standardization must be done at least every three months. 1 mL 0.1000N solution is equal to 5.00 mg CaCO_3 . Calculate normality using:

$$N = \frac{A \times B}{53.00 \times C}$$

where: A = g Na_2CO_3 weighed into 1 liter

B = mL Na_2CO_3 solution

C = mL acid used to inflection point

- 10.4 Standard Acid (sulfuric or hydrochloric), 0.02N (low titrant): Dilute 200.0 mL of 0.1000 N Standard Acid to 1 liter with distilled water. Standardize by potentiometric titration of 15.0 mL 0.05N Na_2CO_3 solution every three months as above. 1 mL 0.020N solution is equal to 1.00 mg CaCO_3 .

11.0 Sample Collection, Preservation, Shipment, and Storage

- 11.1 No preservation necessary except to keep chilled to 4°C until sample is analyzed. Do not open sample bottle until analysis.
- 11.2 The holding time for these samples is 14 days.

12.0 Quality Control

- 12.1 Run a blank spike (BS) for each batch of samples (maximum of 20 samples per day). If the BS does not fall in the range of 80 to 120%, corrective action must be taken to find the problem and correct it.
- 12.2 Run a preparation blank (BLK) for each batch of samples (maximum of 20 samples per day). The BLK should be less than the reporting limit.
- 12.3 Analyze a matrix spike (MS) and matrix spike duplicate (MSD) every 20 samples. The percent recoveries on a MS and MSD should be within 75 to 125% and the relative percent difference (RPD) on duplicates should be less than 20%.
- 12.4 The Excel file for calculations is located in "V:\Lab\Wetchem\TESTS\CarbonDioxide Alkalinity\".

- 12.5 Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.

Calculate spikes as follows where everything is in concentration.

$$\% \text{ Recovery} = \frac{\text{Spike} - \text{Sample}}{\text{True Value}} \times 100$$

Relative percent difference is calculated as follows, with everything in concentration:

$$\text{RPD} = \frac{\text{Higher Concentration} - \text{Lower Concentration}}{\text{Average of Concentrations}} \times 100$$

13.0 Calibration and Standardization

- 13.1 Quality Systems **SOP QS08** "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

14.0 Procedure

- 14.1 Write down time test started.
- 14.2 Fill 10 mL microburette with Standard Acid.
- 14.3 Pick titrant according to estimated total alkalinity. For example, a drinking water or groundwater sample would probably use the 0.020 N titrant and a wastewater sample would probably use the 0.10 N titrant. Historical data is very useful for this.

A sample size of 25 mL is usually appropriate. If you use less than 1 mL of your high titrant, then you must titrate a new sample using low titrant. Using less than 1 mL of your low titrant is valid. When the samples are soils a 12 gram portion diluted to 120 mLs is used.

- 14.4 Sample size should be such that a sufficiently large volume of titrant is used (1 to 10 mL titrant).
- 14.5 Standardize and calibrate the pH meter according to laboratory procedures below. Using the Orion 420A pH meter. The calibration buffer readings must be within ± 0.05 pH units of the true value. If not, recalibration is necessary. Record this information in the appropriate log book. If automatic temperature compensation is not provided, make titration at $25 \pm 2^\circ\text{C}$. Check the buffer every 3 hours after calibration. The reading should be within ± 0.20 pH units.

14.5.1 Initial calibration (2 point)

The Orion 420A meter is capable of both autocalibration and manual calibration. We calibrate manually.

1. Turn the power on. It will display a quick check mode and then the reading will stabilize. The meter should be in the pH mode, if not, press the "mode" key until the pH mode indicator is displayed.
2. Uncover the hole on the upper part of the electrode (If electrode is equipped with one). This allows a uniform flow of the filling solution. (It should be kept covered when not in use.)

3. Rinse the electrode with deionized water and blot dry with a kimwipe (do not wipe the electrode).
4. The meter must be calibrated prior to use with two pH buffer standards (4 and 7). Place both the ATC and the pH electrode in pH 7 buffer first, with stirring bar turning. Make sure the electrodes are not touching the bottom or sides of the beaker. Buffers must be freshly poured up daily from their 2 to 4 Liter storage containers into disposable beakers and be at room temperature.
5. Press the "2nd" key, followed by the "cal" key. It will display the time and date of the last calibration and then "P1" will be displayed in the lower field -- this means it is ready to read the first calibration standard.
6. Wait for a stable pH display and the meter will say "ready". Press the "no" key. The first digit will start flashing. Scroll up or down using the arrow-head keys until the correct value appears in the first digit (it will be 7 for the 7.00 buffer) then press "yes". Continue in the same manner until all the digits have been correctly entered, then press "yes" to enter the new value. The display will remain frozen for a few seconds, then "P2" will be displayed which means that the meter is ready to read the second calibration standard.
7. Rinse the electrode and place in the pH 4 buffer with stirring bar turning. Wait for a stable pH reading and then enter the correct value as you did for the 7 buffer.
8. After both points have been calibrated, the electrode slope will be displayed in the main field. The slope should be between 92 and 102. (Record the slope in the calibration logbook and on the data sheet.) If it is not within this boundary you should inspect the electrode and meter and recalibrate with fresh buffers. If the slope is still out of range, check the manual for troubleshooting.
9. The meter will automatically advance to the "measure" mode.

- 14.6 Carefully pour 25 mLs into disposable beaker by gently pouring down the side of the vessel so as to have the least aeration to sample as possible. Place a small magnetic stirring bar in vessel and start magnetic stirrer at medium to slow stirring.

Note 1: When soils are being analyzed 12 grams is weighed into a 120 mL bottle and diluted to 120 mLs. Place in the shaker for one hour. Mix the sample well and use 25 mLs to analyze. Make sure you get a representative sample for analysis.

Note2: Where sample volume is adequate and low titrant is used (alkalinity concentration is less than 20 mg CaCO₃/L), a sample volume of 100 to 200 mL should be used and titration should be performed using a 10 mL microburette.

- 14.7 Place pH probe (which has been rinsed with DI water and patted dry with a Kimwipe) in the sample such that the probe tip is not touching the sides or bottom of the flask or beaker. If the probe has a protective cover, this is not a consideration.

- 14.8 Make sure there are no air bubbles at the bottom of filled burette. Wipe tip of burette so that no extra drops are clinging to it. Place tip of burette into mouth of vessel so that it is above the surface of the sample but is not touching the sides of the flask and drops can go nowhere but into the sample (e.g., drops from burette are not going onto pH probe or walls of flask but directly into sample).

- 14.9 Titrate a blank and an LCS first. This will let you know that the normalities of titrant are correct. If the result is out of the acceptable range of the LCS, run a duplicate LCS. If still out of range,

find another second source and if still incorrect, restandardize titrant. First double-check titrant normality.

- 14.10 Record sample pH after reading is stable for 5 to 10 seconds.
 - 14.11 Titrate sample to pH 4.5. This must be done slowly so as not to miss the exact pH. Record titrant volume.
 - 14.12 The minimum titrant volume to be employed using high titrant is 1 mL. If high titrant is being used, go to low titrant; if low titrant doesn't work, use more sample. Be aware of sample volume that may be needed for other analyses. Do not dilute.
 - 14.13 When titrating the sample, be sure to allow time for the pH to equilibrate so that the inflection point will not be passed.
 - 14.14 Potentiometric titration of low alkalinity
 - 14.14.1 For alkalinity of < 20 mg CaCO₃/L titrate 100 – 200 mL as above using a 10 mL microburet and 0.02 N acid solution.
 - 14.14.2 Stop titration at pH in range of 4.3-4.7, record volume and exact pH. Very carefully add titrant to lower pH exactly 0.3 pH units and record volume.
 - 14.14.3 If the pH of the sample is above 8.3, the sample needs to be titrated for phenolphthalein alkalinity, first check original pH. If it is not above 8.3, the phenolphthalein result will be below the minimum detection limit. If the original pH is above 8.3, titrate sample as in above procedure but down to 8.3 instead of 4.5, and record titrant volume in box A on the alkalinity bench sheet. Then proceed with the regular procedure titrating to pH 4.5, and recording this result in box B on the alkalinity bench sheet, then carefully titrate exactly 0.3 pH units lower to pH 4.2 and record titrant volume at this level, in box C on the alkalinity bench sheet.
 - 14.15 Potentiometric titration of high alkalinity: Use a sufficiently large volume of titrant (>20 mL in a 50 mL buret) to obtain good precision while keeping volume low enough to permit sharp endpoint.
 - 14.15.1 For >1000 mg CaCO₃/L use 0.1 N titrant
 - A. For alkalinity of > 1000 mg CaCO₃/L, titrate 25 – 50 mL as above using a 50 mL burette and 0.10 N acid solution.
 - B. Stop titration at pH in range of 4.5, record volume and exact pH. See note above.
- 15.0 Data Analysis and Calculations**
- 15.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.
 - 15.2 The detection limit is 1.0 mg/L CaCO₃.

- 15.3 Potentiometric titration to pH 4.5 (high alkalinity)
 Total Alkalinity mg/ L CaCO₃ = $\frac{A \times N \times 50,000}{\text{mL of Sample}}$

Where: A = mL Standard Acid to pH 4.5
 N = Normality Standard Acid

Potentiometric titration of low alkalinity = T below

- 15.4 Method 2320B calculations
 Use the following notation in below calculations:

P = Phenolphthalein alkalinity
 T = Total alkalinity

$$P = \frac{A \times N \times 50,000}{\text{mL of Sample}}$$

$$T = \frac{(2B-C) \times N \times 50,000}{\text{mL of Sample}}$$

If P	= 0	Carbonate	= 0	Bicarbonate	= T
If P	< 1/2T	Carbonate	= 2P	Bicarbonate	= T-2P
If P	= 1/2T	Carbonate	= 2P	Bicarbonate	= 0
If P	> 1/2T	Carbonate	= 2(T-P)	Bicarbonate	= 0
If P	= T	Carbonate	= 0	Bicarbonate	= 0

Where: A = mL titrant to pH 8.3
 B = mL titrant to pH 4.5
 C = mL titrant to pH 4.2
 N = normality Standard Acid

16.0 Method Performance

- 16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

- 16.2 Forty analysts in seventeen laboratories analyzed synthetic water samples containing increments of bicarbonate, with the following results:

Increment as Alkalinity mg/L, CaCO ₃	Precision as Standard Deviation mg/L, CaCO ₃	Accuracy as	
		Bias, %	Bias, mg/L, CaCO ₃
8	1.27	+10.61	+0.85
9	1.14	+22.29	+2.0
113	5.28	-8.19	-9.3
119	5.36	-7.42	-8.8

17.0 Pollution Prevention

- 17.1 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability. Please see Waste Disposal SOP QS14, for instruction of proper disposal of waste generated from this area.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

- 18.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

- 19.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.
- 19.2 If the preparation blank is higher than the reporting limit, all samples less than ten times the concentration of the blank must be reanalyzed.
- 19.2.1 If the blank spike (BS) is out of the range of 80 to 120%, and the % recovery is high (higher than 120%), only sample concentrations less than the reporting limit are acceptable data. Otherwise, all data with concentrations above the method detection limit must be reanalyzed with an BS in the range of 80 to 120%. If the BS is low (less than 80%), all samples must be reanalyzed.

20.0 Waste Management

- 20.1 Please see Waste Disposal SOP QS14, for instruction of proper disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

21.0 References

- 21.1 Standard Methods for the Examination of Water and Wastewater 22nd Edition, method 2320B, editorial revisions -2011 (SM 2320 B-2011).

22.0 Tables, Diagrams, Flowcharts and Validation Data

- 22.1 Table 1, all applicable parameters with the applicable RL.
- 22.2 Table 2, for all technical methods, should always be the QA/QC summary table.
- 22.2 Table 3, Technical Completeness / Accuracy Checklist
- 22.4 Table 4, Data Reviewers Checklist
- 22.5 Validation data would be actual documentation (eg: a pdf email from a regulator explaining the approach to a method, etc.) or a side by side study performed to reach to our approach on how we handle the method.

DEFINITIONS

°C - degrees centigrade
CRDL - contract required detection limit
%D - percent difference
DOD - Department of Defense
BS - blank spike or laboratory control sample
BSD - blank spike duplicate or laboratory control sample duplicate
MDL - method detection limit
mg/KG - milligrams per kilogram
mg/L - milligrams per liter
μL - microliter
μm - micrometer
ml - milliliter
mm - millimeter
MS - matrix spike
MSD - matrix spike duplicate
%RSD - percent relative standard deviation
SOP - standard operating procedure

Refer to SOP QS08 for further definitions.

TABLE 1 – Analytes, Reporting Limit (RL), & Low Calibration Standard (Units mg CaCO₃/L)

Parameter	RL	LowCal
TOTAL ALKALINITY , CARBONATE, BICARBONATE	1.0	NA

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Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
BLK	One per prep batch	<ul style="list-style-type: none"> Must be less than the LOD 	<ul style="list-style-type: none"> Re-analysis to confirm the positive value Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action Re-prep of samples associated with the MB NCR will be required for data reported Final Report data flagging will be required
BS	One per prep batch	80 to 120%	<ul style="list-style-type: none"> Follow guidelines from SOP QS05
BSD	One per prep batch, when MS/MSD not included.	80 to 120% and 20% RPD	<ul style="list-style-type: none"> Follow guidelines from SOP QS05
MS	One per prep batch, if sample volume available.	75 to 125%	<ul style="list-style-type: none"> Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	75 to 125% and 20% RPD	<ul style="list-style-type: none"> Follow guidelines from SOP QS05
DOC Study	<ul style="list-style-type: none"> Initially per analyst prior to reporting data Annually 	<ul style="list-style-type: none"> Average percent recovery should be between 80-120%, with a 20% standard deviation. 	<ul style="list-style-type: none"> Re-prepare and / or re-analysis
MDL Study	Once per year as required for state certifications.	<ul style="list-style-type: none"> Calculated value must be greater than 10% of the Spike Level Calculated value must be less than the Spike level 	<ul style="list-style-type: none"> Re-prepare and / or re-analysis Follow guidelines from SOP QS05

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
5. If the data was entered into LIMS manually, was a check of all entered values performed
6. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
7. Were proper data qualifiers applied to the data in LIMS
8. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

Alkalinity, Carbonate, Bicarbonate Data 2 nd check
<input type="checkbox"/> Analyst authorization
<input type="checkbox"/> All general information complete
<input type="checkbox"/> Base MDL
<input type="checkbox"/> Correct Units
<input type="checkbox"/> Corrections crossed out & initialed
<input type="checkbox"/> Necessary NCR's attached
<input type="checkbox"/> BLK/BS per day & per 20 samples & in control
<input type="checkbox"/> TV's & sources indicated
<input type="checkbox"/> Titrant standardized Quarterly
<input type="checkbox"/> Titrant normality recorded
<input type="checkbox"/> 10% check of calculation
<input type="checkbox"/> MS/MSD per 20 samples
<input type="checkbox"/> Samples noted when <DL
<input type="checkbox"/> BS prep. indicated &/or calculation shown
<input type="checkbox"/> Holding time met
<input type="checkbox"/> Problems discussed with manager
<input type="checkbox"/> Data entered in LIMS correctly
<input type="checkbox"/> Batch sheet completed accurately
<input type="checkbox"/> Additional information needed for reports:
2 nd checked by/date:

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

INORGANICS: SOP 145

REVISION #: 10

EFFECTIVE DATE: 20130806

**DETERMINATION OF INORGANIC ANIONS IN WATER BY ION CHROMATOGRAPH USING
THE DIONEX dx-500 ION CHROMATOGRAPH WITH HYDROXIDE ELUENT AND DIONEX
AS18 COLUMN**

References:

USEPA METHOD 300.0/ SW846 Method 9056/A

APPROVALS:

Lab Director:



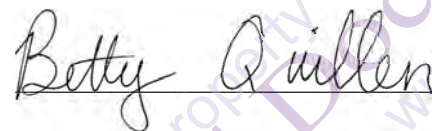
Date: 08/06/2013

Data Quality Manager:



Date: 08/06/2013

Section Supervisor:



Date: 08/06/2013

Changes Summary:

Revision 10, 20130806

- Updated Table 3, Analyst Data Review Checklist to include manual integrations checked by a second reviewer.
- Defined standard expiration dates for vendor stocks, intermediate standards and dilute working standards in section VII and moved Traceability to this section.

Revision 09, 08/20/2012

- QS SOP references added as appropriate.
- Anion F-Table from DoD QSM added to appendix.
- Checklist updated to require LCR recovery calculation on the raw data and add space to record sample reflecting full recalculation.
- Reference to SOP145A added for soils/sediments.

Revision 08 Date: 05/16/2011

- This is an update from SOP revision 07 dated 03//25/10
- Frequency requirements for MDL study, calibration curve and linear calibration range verification added.
- References updated to include method 9056A
- Linear calibration range verification criteria added.
- Addressed method 9056 request for dilution with eluent.

Revision Date: 03/25/2010

- The SOP has been reviewed for accuracy and completeness.
- All references to analysis of ortho-phosphorus by this method have been removed.

DETERMINATION OF INORGANIC ANIONS IN WATER BY ION CHROMATOGRAPH USING THE DIONEX dx-500 ION CHROMATOGRAPH WITH HYDROXIDE ELUENT AND DIONEX AS18 COLUMN

References:

USEPA METHOD 300.0/ SW846 Method 9056/A

I. SCOPE AND APPLICATION:

1. This method covers the determination of the following inorganic common anions in reagent water, surface water, ground water, and other aqueous matrixes. This method may be applied to soils/sediments after creating a leachate as per SOP 145A.

PART A.--Common Anions

Chloride	Nitrate	Fluoride
Nitrite	Bromide	Sulfate

2. Single laboratory Method Detection Limit for the above analytes is listed in Tables 1A, 1B and 1C from method 300.0. The MDL for a specific matrix may differ from those listed, depending upon the nature of the sample and the specific instrumentation employed.
 - A. In order to achieve comparable detection limits, an ion chromatographic system must utilize suppressed conductivity detection, be properly maintained and must be capable of yielding a baseline with no more than a 5 nS noise/drift per minute of monitored response over the background conductivity.
3. This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.
4. When the method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of a laboratory fortified matrix sample covering the anions of interest. The fortification procedure is described in the Quality Control section.
5. Users of the method data should state the data-quality objectives prior to analysis. Analyst using this method must demonstrate the ability to generate acceptable results with the method, using the procedures described in the Quality Control section.

II. SUMMARY OF METHOD

1. A small volume of sample, 50 uL for Part A is introduced into an ion chromatograph (IC). The anions of interest are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.

III. DEFINITIONS

Laboratory Quality System SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

1. **ANALYSIS BATCH** -- A group of no more than 20 field samples (Field sample analyses include only those samples derived from a field sample matrix. These include the initial and duplicate field samples as well as all Laboratory Fortified Sample Matrices (MS/MSD)). The analysis batch must include an Initial Calibration Check Standard (CCV), and End Calibration Check Standard (ending CCV), Laboratory Reagent Blank (BLK), and a Laboratory Fortified Blank (BS). Within an ANALYSIS BATCH, for every group of ten field samples at least one Laboratory Fortified Matrix (MS) and either a Field Duplicate or a

Laboratory Duplicate must be analyzed after the tenth field sample analysis. MSD does not count as a laboratory duplicate for anions.

2. **CALIBRATION STANDARD (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions and the surrogate analyte. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
 - A. **INITIAL CALIBRATION STANDARDS** -- A series of CAL solutions used to initially establish instrument calibration and develop calibration curves for individual target anions. Complete calibration required every 6 months.
 - B. **INITIAL CALIBRATION CHECK STANDARD** -- An individual CAL solution, analyzed initially, prior to any sample analysis, which verifies previously established calibration curves.
 - C. **CONTINUING CALIBRATION CHECK STANDARD** -- An individual CAL solution which is analyzed after every tenth field sample analyses which verifies the previously established calibration curves and confirms accurate analyte quantitation for the previous ten field samples analyzed.
 - D. **END CALIBRATION CHECK STANDARD** -- An individual CAL solution which is analyzed after the last field sample analyses which verifies the previously established calibration curves and confirms accurate analyte quantitation for all field samples analyzed since the last continuing calibration check.
3. **FIELD DUPLICATES (FD)** -- Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout the field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.
4. **INSTRUMENT PERFORMANCE CHECK SOLUTION (ICV)** -- A solution of one or more method analytes, surrogates or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.
5. **LABORATORY DUPLICATE (DUP)** -- Two sample aliquots, taken in the laboratory from a single sample bottle, and analyzed separately with identical procedures. Analyses of sample and DUP1 indicate precision associated specifically with the laboratory procedures, removing any associated variables attributed by sample collection, preservation, or storage procedures.
6. **LABORATORY FORTIFIED BLANK (BS)** -- An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.
7. **LABORATORY FORTIFIED SAMPLE MATRIX (MS)** -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS and or MSD are analyzed exactly like a sample, and their purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.
8. **LABORATORY REAGENT BLANK (BLK)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The BLK is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
9. **LINEAR CALIBRATION RANGE (LCR)** -- The concentration range over which the instrument response is linear. Verification at the high standard required every 6 months.

10. **MATERIAL SAFETY DATA SHEET (MSDS)** -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.
11. **METHOD DETECTION LIMIT (MDL)** -- The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero. Method recommends MDL every 6 months. Annual MDL performed with quarterly verification at low standard.
12. **MINIMUM REPORTING LEVEL (MRL)** -- The minimum concentration that can be reported for an anion in a sample following analysis. This defined concentration can be no lower than the concentration of the lowest calibration standard and can only be used if acceptable quality control criteria for this standard are met.
13. **PERFORMANCE EVALUATION SAMPLE (PE)** -- A certified solution of method analytes whose concentration is unknown to the analyst. Often, an aliquot of this solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are used to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst.
14. **QUALITY CONTROL SAMPLE (QCS)** -- A solution of method analytes of known concentrations that is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.
15. **STOCK STANDARD SOLUTION (SSS)** -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

IV. INTERFERENCES

1. Interferences can be divided into three different categories: **direct chromatographic coelution**, where an analyte response is observed at very nearly the same retention time as the target anion; **concentration dependant coelution**, which is observed when the response of higher than typical concentrations of the neighboring peak overlap into the retention window of the target anion; and, **ionic character displacement**, where retention times may significantly shift due to the influence of high ionic strength matrices (high mineral content or hardness) overloading the exchange sites in the column and significantly shortening target analytes' retention times.
 - A. A direct chromatographic coelution may be solved by changing columns, eluant strength, modifying the eluant with organic solvents (if compatible with IC columns), changing the detection systems, or selective removal of the interference with pretreatment. Sample dilution will have little to no effect. The analyst must verify that these changes do not negatively affect performance by repeating and passing all the criteria in the Quality Control Section.
 - B. Sample dilution may resolve some of the difficulties if the interference is the result of either concentration dependant coelution or ionic character displacement, but it must be clarified that sample dilution will alter your Minimum Reporting Limit (MRL) by a proportion equivalent to that of the dilution. Therefore, careful consideration of project objectives should be given prior to performing such a dilution. An alternative to sample dilution, may be dilution of the eluant.
 - C. Pretreatment cartridges can be effective as a means to eliminate certain matrix interferences. Prior to using any pretreatment, the analyst should be aware that all **instrument calibration standards must be pretreated in exactly the same manner** as the pretreated unknown field samples. The need for these cartridges has been greatly reduced with recent advances in high capacity anion exchange columns.

1. Extreme caution should be exercised in using these pretreatment cartridges. Artifacts are known to leach from certain cartridges, which can foul the guard, and analytical columns causing loss of column capacity indicated by shortened retention times and irreproducible results. Frequently compare your calibration standard chromatograms to those of the column test chromatogram (received when the column was purchased) to insure proper separation and similar response ratios between the target analytes is observed.
- D. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in an ion chromatogram. These interferences can lead to false positive results for target analytes as well as reduced detection limits as a consequence of elevated baseline noise.
- E. Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.
- F. Any anion that is only weakly retained by the column may elute in the retention time window of fluoride and potentially interfere. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant; however, it is the responsibility of the analyst to generate precision and accuracy information in each sample matrix.
- G. Close attention should be given to the potential for carry over peaks from one analysis which will effect the proper detection of analytes of interest in a second, subsequent analysis. The elution of nitrate (retention time of ~9.0 min.) indicates the end of a chromatographic run. A run time of 12 minutes is recommended to allow for the proper elution of any potentially interferrant late peaks. It is the responsibility of the analyst to confirm that no late eluting peaks have carried over into a subsequent analysis thereby compromising the integrity of the analytical results.

V. SAFETY

1. Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.
2. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of latex gloves and lab coats is highly recommended.
3. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
4. MSDS sheets are available for all reagents and standards that have been purchased. These are located on the bookshelves in the Data Quality Manager's office.

VI. EQUIPMENT AND SUPPLIES

1. Ion Chromatograph (IC) – Analytical system complete with eluant generator, an ion chromatographic pump, injection valves, both guard and analytical separator columns, suppressor, conductivity detector, and computer based data acquisition system. Dionex DX-500 or equivalent. (See letter from EPA to Dionex on discussion of alternate hydroxide eluant for anions. Also since hydroxide eluant cannot be run on the traditional column Dionex AS18, 4mm (P/N 060549) or equivalent should be used.
 - a. Anion guard column--Dionex Ion Pac AG18 4mm (P/N 060551), or equivalent. This column functions as a protector of the separator column. If omitted from the system, the retention times will be shorter.

- b. Anion separator column--Dionex Ion Pac AS18, 4mm (P/N 060549), or equivalent. An optional column (2mm or 4 mm) may be used if comparable resolution of peaks is obtained, and the quality control requirements can be met.
 - i. When a 4 mm column is employed, the injection volume should be 50 μ L.
 - ii. Comparable results can be attained using the Dionex, AS17, 4 mm column.
 2. Anion suppressor device--The data presented in this method were generated using an Ultra 4 mm Dionex Anion Self Regenerating Suppressor (ASRS, P/N 53946). An equivalent suppressor device may be utilized provided comparable conductivity detection limits are achieved and adequate baseline stability is attained as measured by a combined baseline drift/noise of no more than 5 nS per minute over the background conductivity. Proper suppressor performance is essential to analytical data reproducibility and sensitivity of the conductivity detector.
 - a. The ASRS was set to perform electrolytic suppression at a current setting of 300 ma using the external water mode. External water was delivered to the suppressor directly from a pressurized source at a flow rate of 5 mL/min. It should be noted that while Empirical Laboratories has the suppressor currently set at 300 mA, no external water is being used at this time.
 3. Detector--Conductivity cell (Dionex CD20, or equivalent) capable of providing data as required in the Quality Control section of this SOP.
 4. Data Acquisition System--The Dionex Peaknet Data Chromatography Software version 5.2 or equivalent is used by Empirical Laboratories.
 5. Analytical balance--Mettler Used to accurately weigh target analyte salt for stock standard preparation (± 0.1 mg sensitivity).
 6. Micro beakers -- Plastic, disposable - used during sample preparation.
 7. Syringes--Plastic, disposable, 10 mL - used during sample preparation.
 8. Eppendorfs with variable settings- 1mL and 5 mL. Must be calibrated quarterly.
 9. Bottles -- High density polyethylene (HDPE) or glass, amber or clear, 30 mL, 125 mL, 250 mL. For sampling and storage of calibration solutions.
 10. Particulate filters-- 0.45 micron syringe filters, specifically designed for IC applications (Gelman IC Acrodisc, PN 4485, or equivalent). These cartridges are used to remove particulates from the sample matrix while loading the sample manually or if the autosampler employed does not filter the sample during loading.
- NOTE: See method for several types of pretreatment cartridges that are available and may be useful depending on the matrices of the samples normally processed.
11. Autosampler PolyVials 5-mL size, with filtercaps, 250 each --Dionex cat log # 38141.
 12. Shaker for use when extracting soil samples.
 13. Centrifuge to aid in separation after extraction.
 14. Centrifuge tubes--50 mL capacity

VII. REAGENTS AND STANDARDS

The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method. **All reagents shall be made from ACS reagent grade chemicals. All reagents used for distillation and analysis are entered into Element. These reagents are added to the batch sheet when the samples are batched to ensure traceability of the reagents used to the associated samples.**

1. Reagent water-- Distilled or deionized water 17.8 Mohm or better, free of anions of interest. Water should contain particles no larger than 0.20 microns.
2. A system or apparatus which automatically generates the hydroxide eluant (Dionex EG40, or equivalent) is an acceptable alternative to physically preparing the hydroxide eluant.
3. Stock standard solutions, 1000 mg/L (1mg/mL): Stock standard solutions are purchased as certified solutions from selected vendors. Expiration dates are assigned as identified by the vendor.

- Intermediate standard solutions: Prepared by dilution of stock standard solutions and assigned expiration date of 3 months from preparation.
- Dilute working standards: Prepared by dilution of intermediate standard solutions and assigned expiration date of 2 days from preparation.

Traceability

A record shall be maintained on all reference materials within Element. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in Element as well as on the container's label.

All working standards made from reference materials shall be labeled with a unique Element ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date, date opened, and the logbook where information is recorded. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded within Element. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

VIII. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

- Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.
- Sample preservation and holding times for the anions that can be determined by this method are as follows:

PART A: Common Anions

<u>Analyte</u>	<u>Preservation</u>	<u>Holding Time</u>
Bromide	None required	28 days
Chloride	None required	28 days
Fluoride	None required	28 days
Nitrate-N	Cool to 4 °C	48 hours
Nitrite-N	Cool to 4 °C	48 hours
Sulfate	Cool to 4 °C	28 days

- When collecting a sample from a treatment plant employing chlorine dioxide, the sample must be sparged with an inert gas (helium, argon, nitrogen) prior to addition of the EDA preservative at time of sample collection.

IX. QUALITY CONTROL

Quality Systems SOP QS08 "Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

- The laboratory is required to operate a formal quality control (QC) program. The requirements of this program consist of an initial demonstration of laboratory performance, and subsequent analysis in each analysis batch of a Laboratory Reagent Blank, Laboratory Fortified Blank, Instrument Performance Check Standard, calibration check standards, Laboratory Fortified Sample Matrices (LFM) and either Field, Laboratory or LFM duplicate sample analyses. This section details the specific requirements for each of these QC parameters. The laboratory is required to maintain performance records that define the quality of the data that are generated.

2. INITIAL DEMONSTRATION OF PERFORMANCE

- A. The initial demonstration of performance is used to characterize instrument performance (determination of accuracy through the analysis of the QCS) and laboratory performance (determination of MDLs) prior to performing analysis by this method.
- B. Quality Control Sample (QCS) – When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. If the determined concentrations are not within $\pm 10\%$ of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.
- C. Method Detection Limit (MDL)—MDLs are established for all analytes, using reagent water (blank) fortified at a concentration of three to five times the estimated instrument detection limit. To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method over at least three separate days. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$\text{MDL} = (t) \times (S)$$

Where,

t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates].

S = standard deviation of the replicate analyses.

- D. MDLs should be generated when a new operator begins work or whenever there is a significant change in the background, or instrument response. MDL check samples are used in connection with confirming that the MDL determined is legitimate and to monitor the instrument sensitivity periodically. MDL checks are analyzed whenever a new MDL is generated and at a minimum quarterly to monitor for any shifts in sensitivity. Method recommends MDLs every 6 months but Empirical performs annually with quarterly verifications.

3. ASSESSING LABORATORY PERFORMANCE

- A. Laboratory Reagent Blank (BLK) – The laboratory must analyze at least one LRB with each analysis batch. Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL (**For DOD QSM no analytes detected $\geq \frac{1}{2}$ RL or for common lab contaminants no analyte detected \geq RL**) indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis.
- B. Laboratory Fortified Blank (BS)—The BS should be prepared at concentrations similar to those expected in the field samples and ideally at the same concentration used to prepare the MS/MSD. Calculate accuracy as percent recovery. If the recovery of any analyte falls outside the required concentration dependant control limits that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.
 - i. Control Limits for the BS are 90 to 110%.
 - ii. The laboratory uses the BS to assess laboratory performance against the required control limits listed in the QC section. When sufficient internal performance data becomes available (usually a minimum of 20-30 analyses), optional control limits can be developed from the percent mean recovery (x)

and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

$$\text{UPPER CONTROL LIMIT} = \bar{x} + 3S$$

$$\text{LOWER CONTROL LIMIT} = \bar{x} - 3S$$

The optional control limits must be equal to or better than those listed in the QC section ($\pm 10\%$). After each five to ten new recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also, the standard deviation (S) data should be used to establish an on-going precision statement for the level of concentrations monitored. These data must be on file and be available for review.

- i. Instrument Performance Check Solution (ICV) – The Initial Calibration Check Standard is to be evaluated as the instrument performance check solution in order to confirm proper instrument performance. The acceptable limits for this standard is 90 to 110%. Small variations in retention time can be anticipated when a new solution of eluant (or when the KOH cartridge is changed) is prepared but if shifts of more than 2% are observed in the IPC retention time, some type of instrument problem is present. Potential problems improperly prepared eluant, erroneous method parameters programmed such as flow rate or some other system problem. The chromatographic profile (elution order) of the target anions following an ion chromatographic analysis should closely replicate the profile displayed in the test chromatogram that was shipped when the column was purchased. As a column ages, it is normal to see a gradual shift and shortening of retention times, but if after several years of use, extensive use over less than a year, or use with harsh samples, this retention time has noticeably shifted to any less than 80% of the original recorded value, the column may require cleaning or replacement. Particularly if resolution problems are beginning to become common between previously resolved peaks. A laboratory must retain a historic record of retention times for all the target anions in the ICV to provide evidence of an analytical column's vitality.

4. ASSESSING ANALYTE RECOVERY AND DATA QUALITY

- A. Laboratory Fortified Sample Matrix (MS) – The laboratory adds a known amount of analyte to a minimum of 10% of the field samples within an analysis batch. The MS sample is prepared from a sample matrix which has been analyzed prior to fortification. The analyte concentration must be high enough to be detected above the original sample and should adhere to the QC requirements. It is recommended that the solutions used to fortify the MS be prepared from the same stocks used to prepare the calibration standards and not from external source stocks. This will remove the bias contributed by an externally prepared stock and focus on any potential bias introduced by the field sample matrix.

- i. If the fortified concentration is less than the observed background concentration of the unfortified matrix, the recovery should not be calculated. This is due to the difficulty in calculating accurate recoveries of the fortified concentration when the native sample concentration is so high.
- ii. The MS should be prepared at concentrations no greater than five times the highest concentration observed in any field sample. If no analyte is observed in any field sample, the MS must be fortified no greater than five times the lowest calibration level which as outlined in this method is the

minimum reported level (MRL). For example, if chloride is not detected in any field samples above the lowest calibrations standard concentration of 5.00 ug/L, the highest MS fortified concentration allowed is 25.0 ug/L.

- iii. Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample. Percent recovery should be calculated using the following equation:

$$R = \frac{C_s - C}{S} \times 100$$

where, R = percent recovery.

C_s = fortified sample concentration

C = sample background concentration

S = concentration equivalent of analyte added to sample.

- iv. Until sufficient data becomes available (usually a minimum of 20 to 30 analysis), assess laboratory performance against recovery limits of 80 to 120%. When sufficient internal performance data becomes available develop control limits from percent mean recovery and the standard deviation of the mean recovery. The optional control limits must be equal to or better than the required control limits of 80 –120%.
- v. If the recovery of any analyte falls outside the designated LFM recovery range and the performance for that analyte is shown to be in control, the recovery problem encountered with the LFM is judged to be matrix induced and the results for that sample and the LFM are reported with a “matrix induced bias” qualifier.

B. FIELD OR LABORATORY DUPLICATES –Analyze either a field, matrix spike duplicate or a laboratory duplicate for a minimum of 10% of the collected field samples or at least one with every analysis batch, whichever is greater. The sample matrix selected for this duplicate analysis must contain measurable concentrations of the target anions in order to establish the precision of the analysis set and insure the quality of the data. If none of the samples within an analysis batch have measurable concentrations, the LFM should be employed as a laboratory duplicate.

- i. Calculate the relative percent difference (RPD) of the initial quantitated concentration (I_c) and duplicate quantitated concentration (D_c) using the following formula,

$$RPD = \frac{(I_c - D_c)}{[(I_c + D_c)/2]} \times 100$$

- ii. Duplicate analysis acceptance criteria

<u>Concentration range</u>	<u>RPD Limits</u>
MRL to 10xMRL	± 20%
10xMRL to highest calibration level	± 10%

- iii. If the RPD fails to meet these criteria, the samples must be reported with a qualifier identifying the sample analysis result as yielding a poor duplicate analysis RPD. This should not be a chronic problem and if it frequently recurs (>20% of duplicate analyses) it indicates a problem with the instrument or individual technique.
- C. Where reference materials are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.
- D. In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options, such as the use of different columns, injection volumes, and/or eluants, to improve the separations or lower the cost of measurements. Each time such modifications to the method are made, the analyst is required to repeat the procedure in the QC section and adhere to the condition of baseline stability.
- E. The laboratory adopts additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the client and the nature of the samples. Whenever possible, the laboratory performs analysis of quality control check samples and participate in relevant performance evaluation sample studies.

5. **CALIBRATION AND STANDARDIZATION**

Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization

- A. Establish ion chromatographic operating parameters equivalent to those indicated in Tables 1C for a 4-mm column.
 - i. Estimate the Linear Calibration Range (LCR) – The LCR should cover the expected concentration range of the field samples.
 - ii. For an individual anion calibration curve, a minimum of five calibration standard concentrations is required. Generally, a total of eight calibration concentrations are analyzed to provide five concentrations for each anion.
- B. Prepare the calibration standards by carefully adding measured volumes of one or more stock standards to a volumetric flask and diluting to volume with reagent water. Chloride and sulfate are calibrated from 0.5-200 mg/L; fluoride, nitrate, and nitrite from 0.05-20 mg/L.
- C. Using a 4mm column, inject 50 uL (Part A) of each calibration standard. Tabulate peak area responses against the concentration. The results are used to prepare calibration curves using a linear least squares fit for each analyte. Acceptable calibration curves are confirmed after reviewing the curves for linearity and passing the criteria for the initial calibration check standard. Alternately, if the ratio of response to concentration (response factor) is constant over the LCR (indicated by < 15% relative standard deviation (RSD)), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.
 - i. Peak areas strongly recommended since they have been found to be more consistent, in terms of quantitation, than peak heights. Peak height can tend to be suppressed as a result of high levels of common anions in a given matrix which can compete for exchange sites. Using peak areas, it is the analyst responsibility to review all chromatograms to insure accurate baseline integration of target analyte peaks since poorly drawn baselines will more significantly influence peak areas than peak heights.

- D. Once the calibration curves have been established they must be verified prior to conducting any sample analysis using an initial calibration check standard. This verification must be performed on each analysis day or whenever fresh eluant has been prepared. A continuing calibration check standard must be analyzed after every tenth sample and at the end of the analysis set as an end calibration check standard. The response for the initial, continuing and end calibration check must satisfy the QC criteria of 90 to 110%. If during the analysis set, the response differs by more than the calibration verification criteria, or the retention times shift more than $\pm 5\%$ from the expected values for any analyte, the test must be repeated, using fresh calibration standards. If the results are still outside these criteria, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable calibration check standard must be reanalyzed.
- i. Control limits for calibration verification are 90 to 110%.
 - ii. Linear Calibration Range must be verified every 6 months with a standard at the highest concentration of the calibration curve. Control limits are 90%-110%.
- F. After satisfying the requirements, the levels selected for the other calibration check standards should be varied between a middle calibration level and the highest calibration level.
- G. Software Entry of new curve
- i. Integrate curve data and print results.
 - ii. Open Current method (Go to Peaknet Main Menu, Method) and do a save as the new date of curve.
 - iii. Open the schedule with the curve (Go to PeakNet Main Menu, Schedule). Change the method to the new method that was just created and change the sample type to Calibration Standard 1, 2, 3, etc... and save.
 - iv. Go to main menu, batch, processing, input tab and select the schedule with the curve. Then go to output tab and check update methods from calibration standards and update raw data files. Then press ok and F5. **EXIT, DO NOT SAVE CHANGES.**
 - v. Open new method and click the 5th data processing icon (blue), then click details and go to each component (analyte) (there's a drop down menu) and check areas with the printouts to make sure all data was pulled in correctly.
 - vi. Go through each analyte making sure the correlation is >0.995 . Also the MDL point cannot be used as part of curve so it must be dropped. To drop a point, deselect it and put in comment NOT USED. Also curvefit = Linear; Origin = Include; Calibrate by = Area. Then take the retention times from the middle point of the curve. Go out of details and go to the identification tab, change the retention times and click apply then ok and save.
 - vii. Once changes have been made go to main menu, batch, processing, input tab and choose schedule, then output tab and check update raw data files. **(DO NOT CHECK UPDATE CALIBRATION STANDARDS or it will erase all the changes that were just made).** Then click OK then F5.
 - viii. Go to main menu – optimize, open the QC files that were ran after curve and check. ICV must be $\pm 10\%$.
 - ix. When curve is complete, gather raw data before and after and print out the curve. To do this open a blank EXCEL sheet, then go to main menu – method – open current method, go to each analyte and press ALT PRINT SCREEN then paste into the EXCEL file. A copy of all this will go to the reporting department.

6.

PROCEDURE

- A. Other columns, chromatographic conditions, or detectors may be used if the requirements of the QC section are met.
- B. Check system calibration daily and, if required, recalibrate as necessary – minimum every 6 months.
- C. Sample Preparation
 - i. For refrigerated or samples arriving to the laboratory cold, ensure the samples have come to room temperature prior to conducting sample analysis by allowing the samples to warm on the bench for at least 1 hour.
- D. Using a Luer lock, plastic 5 to 10 mL syringe, withdraw the sample from the micro beaker and attach a 0.45 μ m particulate filter (demonstrated to be free of ionic contaminants) directly to the syringe. Filter the sample into an autosampler vial.
- E. Using a 4 mm column, inject 50 μ L of each sample. Tabulate peak area responses against the concentration. During this procedure, retention times must be recorded. Use the same size loop for standards and samples. Record the resulting peak size in area units. An automated constant volume injection system may also be used.
- F. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.
- G. If the response of a sample analyte exceeds the calibration range, the sample may be diluted with an appropriate amount of reagent water and reanalyzed. If this is not possible then three new calibration concentrations must be employed to create a separate high concentration curve, one standard near the estimated concentration and the other two bracketing around an interval equivalent to $\pm 25\%$ the estimated concentration. The latter procedure involves significantly more time than a simple sample dilution therefore; it is advisable to collect sufficient sample to allow for sample dilution or sample reanalysis, if required. (Note: Method 9056 samples should be diluted with eluent. As the instrument is configured with an eluent generator, this is not applied.)
- H. Shifts in retention time are inversely proportional to concentration. Nitrate, phosphate and sulfate will exhibit the greatest degree of change, although all anions can be affected. In some cases this peak migration may produce poor resolution or make peak identification difficult.
- I. Should more complete resolution be needed between any two coeluting peaks, the eluant can be diluted. This will spread out the run, however, and will cause late eluting anions to be retained even longer. The analysts must verify that this dilution does not negatively affect performance by repeating and passing all the QC criteria.
 - i. Eluant dilution will reduce the overall response of an anion due to chromatographic band broadening which will be evident by shortened and broadened peaks. This will adversely affect the MDLs for each analyte.

7 DATA ANALYSIS AND CALCULATIONS

Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

- A. Prepare a calibration curve for each analyte by plotting instrument response, as peak area, against standard concentration. Compute sample concentration by comparing sample response with the standard curve. If a sample has been diluted, multiply the response by the appropriate dilution factor.
- B. Report ONLY those values that fall between the lowest and the highest calibration standards. Samples with target analyte responses exceeding the highest standard should be diluted and reanalyzed. Samples with target analytes identified but quantitated below

the concentration established by the lowest calibration standard should be reported as below the minimum reporting limit (MRL).

- C. Report results for Part A anions in mg/L.
- D. Report NO_3^- as N

IX. INSTRUMENT INFORMATION

Analyst should confirm the following:

1. **Start Up routine for instrument is as follows:**
 - a. Turn Power on to autosampler, conductivity detector, eluant generator and pump in any order.
 - b. Turn on the Helium gas supply~ 80 to 100. Ensure gas lines to bottles not in use are off. Ensure air supply to the injection valve is on.
 - c. Close the vent valves on sparging bottles and allow head pressure to build for a few moments. DI H₂O bottles should be 7 to 10 psi.
 - d. Open the eluant supply valve(s) for the bottles in use. Check for sputter after eluant flow has started.
 - e. Inject 5 mL of DI water into the pump head and clean the pump head. Also check the frits on the guard column and change if necessary. If necessary to change guard column frits check primary column frits as well.
 - f. Load the auto sampler cartridges and put autosampler into RUN state.
 - g. To vent airlocks, Run eluant with pump on and SRS off, open waste valve-> bottom door top black knob, just for a few seconds and close it.
 - h. Turn on the SRS power. NOTE: NEVER TURN ON THE SRS POWER SUPPLY WITHOUT THE PUMP GOING FIRST!!! Use either LOCAL/DIRECT CONTROL to enter commands at keypads, or REMOTE/DIRECT CONTROL to use the direct control option from the RUN menu within PEAKNET.
 - i. After System has come to equilibrium, load sequence and run.
2. **Shut Down routine for instrument is as follows:**
 - a. If the instrument is not going to be operated for a period of time, run deionized water through the eluant lines for ~ 30 minutes to an hour to rinse the lines.
 - b. Stop the OFF/ON pump and then select SRS-OFF.
 - c. Close gas supply valves and eluant valves on the eluant bottles. Turn off the supply. Is not necessary to vent the eluant bottles.
 - d. Power down the modules in any order.
3. **General Sample Loading and Run Set-up.**
 - a. Enter Peak-net Software from Desktop.
 - b. Loading a Run: Click on Schedule. The headings within the Schedule Editor are SAMPLE, SAMPLE TYPE, LEVEL, METHOD and DATA FILE.
 - i. Name each sample under the SAMPLE heading column. (, CCV, CCB, CRL, BS, BLK, sample #'s, etc.)
 - ii. SAMPLE TYPE is sample unless loading a calibration curve.
 - iii. LEVEL designations are used only when assigned to a calibration curve.
 - iv. Enter method name under METHOD heading. In most cases, date of most recent calibration in Anions Method file will be used.
 - v. Enter the date under the DATA FILE heading. The program will then sequentially assign the data file names based on the date.
 - vi. All other column headings are defaulted to enter "1". Samples requiring dilution should be left at "1" and manual calculation is required.

- vii. To include a command to Shut Down the pump at the end of the run: Name the row following the last sample, Pump Off under the sample heading. It is not necessary to include a vial in the corresponding position in the autosampler. Sample type is Sample, and Method is entered as <pumpoff.met>.

Typical run-log:

1	Blank
2	CCV
3	CCB
4	CRL1
5	CRL2
6	BS1
7	BLK1
8	Sample
9	Sample @ 10X
10	Sample @ 50X
11	Sample
12	Sample
13	Sample
14	CCV
15	CCB
16	Sample
17	Sample
18	Sample
19	Sample
20	Sample
21	Sample
22	Batch #-MS1
23	Batch #-MSD1
24	Batch #- DUP1
25	Sample @ 10X
26	Sample
27	Sample
28	Sample
29	CCV
30	CCB
31	Sample
32	Sample
33	Sample
34	Sample
35	Sample
36	Batch #-MS2
37	Batch #-MSD2
38	Batch #- DUP2
39	Sample
40	Sample
41	Sample
42	Sample
43	CCV
44	CCB

45	Sample
46	Sample
47	Sample
48	CCV
49	CCB
50	pumpoff

- c. Save a schedule under File/Save as, using the date as the title of the Schedule.
- d. When saved, exit out of Schedule Editor.
- e. Load autosample cartridges in the same order as the scheduled run. After putting the cartridges in the autosampler, switch the autosampler to RUN using the Hold/Run button.
- f. Assuming that the Pump is equilibrated with steady eluant baseline/uniform conductivity, enter into the RUN page.
- g. Go to File to Open Method. Open correct method <date> of most recent calibration. This will begin pumping eluant at 1.0 mL/minute and turn on the SRS pump at 300 μ amps voltage.
- h. Next, go to file to Open Schedule. Open newly created schedule for the day.
- i. When Method and Schedule are opened, go to Run and click on Start. The autosampler will inject into the first sample and the run should continue until completion.
- j.

X. POLLUTION PREVENTION

- A. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.
- B. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.
- C. For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

XI. WASTE MANAGEMENT

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

- A. The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Section 14.3 from method 300.1.

XII. CORRECTIVE ACTIONS

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. See **table 2** for specified criteria and appropriate actions.

XIII. SOURCES/REFERENCES:

1. Standard Methods for the Examination of Water and Wastewater, Method 4110B, "Anions by Ion Chromatography", 18th Edition of Standard Methods (1992).
2. Dionex, System DX500 Operation and Maintenance Manual, Dionex Corporation, Sunnyvale, California 94086, 1996.
3. Method Detection Limit (MDL) as described in "Trace Analyses for Wastewater," J. Glaser, D. Foerst, G. McKee, S. Quave, W. Budde, Environmental Science and Technology, Vol. 15, Number 12, page 1426, December, 1981.
4. American Society for Testing and Materials. Test Method for Anions in Water by Chemically – Suppressed Ion Chromatography D4327-91. Annual Book of Standards, Vo. 11.01 (1993).
5. Code of Federal Regulations 40, Ch. 1, Pt. 136, Appendix B; MDL determination.
6. Hautman, D.P. & Bolyard, M. Analysis of Oxyhalide Disinfection By-products and other Anions of Interest in Drinking Water by Ion Chromatography. Jour. Of Chromatog., 602, (1992), 65-74.
7. USEPA Methods 300.0; *Method for Determination of Inorganic Substances*(EPA/600/R-93/100) / *Method for the Determination of Organic and Inorganic Compounds in Drinking Water* (Vol. 1, EPA 815-R-00-014).
8. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 9056.
9. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update IV); Method 9056A.

XIV. TABLES

- 22.1 Table 1, all applicable parameters with applicable RL and lowest calibration standard.
- 22.2 Table 2, 9056 QA/QC summary table.
- 22.3 Table 3, Analyst Data Review Checklist

Table 1

S/W	Method	Analyte	MRL/LOQ	LOD	MDL/DL	Units
Water	300_/9056A	Bromide	0.250	0.125	0.050	mg/L
Water	300_/9056A	Chloride	0.500	0.33	0.170	mg/L
Water	300_/9056A	Fluoride	0.25	0.10	0.0330	mg/L
Water	300_/9056A	Nitrate as N	0.25	0.10	0.0330	mg/L
Water	300_/9056A	Nitrite as N	0.25	0.10	0.0330	mg/L
Water	300_/9056A	Sulfate as SO4	2.5	1.0	0.330	mg/L
Solid	9056A	Bromide	2.5	1.25	0.5	mg/Kg
Solid	9056A	Chloride	5.0	3.3	1.7	mg/Kg
Solid	9056A	Fluoride	2.50	1	0.33	mg/Kg
Solid	9056A	Nitrate as N	2.50	1	0.33	mg/Kg
Solid	9056A	Nitrite as N	2.50	1	0.33	mg/Kg
Solid	9056A	Sulfate as SO4	25.0	10	3.3	mg/Kg

Table 2. Common Anions Analysis (Method 9056)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Quarterly	Detection at established LOD			
LOQ establishment and verification	Quarterly	Recovery within LCS limits at 1x-4x established LOQ.			
Retention time (RT) window width calculated for each analyte	After method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT over a 24-hour period.	NA.	NA.	
Initial calibration (ICAL) for all analytes (minimum three standards and one calibration blank)	ICAL prior to sample analysis repeated every 6 months or as required when ICV exceeds limits.	$r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Linear Calibration Range (LCR)	Every 6 months (with calibration)	All analytes within $\pm 10\%$ of true value and retention times within appropriate windows at highest range.	Validate next lowest standard. Samples exceeding the LCR must be diluted to within range.		
Initial calibration verification (ICV) (second source)	Once after each ICAL, prior to beginning a sample run.	All analytes within $\pm 10\%$ of true value and retention times within appropriate windows.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte	Once per multipoint calibration.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Midrange continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All project analytes within established retention time windows. Within ± 10% of true value.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported	One per preparatory batch.	Recovery ± 20% 9056A Recovery ± 10% EPA 300.0	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix.	Recovery ± 20% If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD)	One per preparatory batch per matrix (see Box D-7).	Recovery ± 20% (see above) RPD ≤ 15% (between MS and MSD).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Sample duplicate (replicate)	One per every 10 samples.	%D ≤ 10% (between sample and sample duplicate).	Correct problem and reanalyze sample and duplicate.	Apply J-flag if sample cannot be rerun or reanalysis does not correct problem.	The data shall be evaluated to determine the source of difference.
Results reported between DL & LOQ	N/A.	N/A.	N/A.	Apply J-flag to all results between DL and LOQ.	

Table 3, ANALYST DATA REVIEW CHECKLIST

Sample Number(s):			
Batch ID(s):	Sequence ID:	Run Date:	NCR#
Method: EPA 300.0/SW846 9056A Anions by Ion Chromatography			
Instrument is a Dionex DX-500 system. Equipped with Guard Column, Analytical Column, Conductivity Suppressor, Conductivity Detector, and Eluant Generator.			

<u>QA/QC Item</u>	<u>Yes</u>	<u>No</u>	<u>NA</u>	<u>2nd Check</u>
1. Were samples analyzed within holding times?				
2. Were initial calibration curve QC criteria met? Curve age <6 months? LCR validated with recoveries shown on results?				
3. Was all continuing calibration criteria in control?				
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)				
5. Did CRL meet control limits?				
6. Did BS, Laboratory Fortified Blank or blank spike meet control limits?				
7. Did MS/MSD meet control limits? Did Duplicate meet control limits?				
8. Was the Blank below the project required detection limits?				
9. Did you return samples back to cold storage immediately after use?				
10. Were samples analyzed for Nitrate (as N) and Nitrite (as N) completed within the 48-hr holding time?				
11. Sample preparation information is correct and complete.				
12. Were all samples filtered through a 0.45µm filter?				
13. Analytical results are correct and complete.				
14. The appropriate SOP's have been used and followed.				
15. Raw data" including all manual integration's have been correctly interpreted and approved by supervisor or approved alternate?				
16. "Special" sample preparation and analytical requirements have been met.				
17. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete.				
18. Sample _____ has complete calculation from raw data to final result reported in Element reflected on raw data.				
19. Are all manual integrations checked by a second reviewer to verify they were performed correctly?				

Comments on any "No" response:

Analyst: _____

Date: _____

Second-Level Review: _____

Date: _____

EMPIRICAL LABORATORIES, LLC (EL)
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 236

REVISION #: 05

EFFECTIVE DATE: 20130711

METHANE, ETHANE, ETHENE IN AQUEOUS SAMPLES
BY MODIFIED RSK-175
(AUTOMATED HEADSPACE)

APPROVALS:

Lab Director:



Date: 07/11/2013

Data Quality Manager:



Date: 07/11/2013

Section Supervisor:



Date: 07/11/2013

Changes Summary

Revision 05, 20130711

- Section 15.2: Updated to include column and row references indicated in calculations.

Revision 04, 20130612

- Section 10.3: indicate gas standards are purchased at concentration of 1.
- Section 11.0: Indicate samples can be received unpreserved or preserved with HCl like VOA samples.
- Section 13.1: correct RSD criteria to indicate must be “less” than 20 percent.
- Section 13.2: add MS/MSD to BS reference.
- Section 14.3.2 and 14.3.3: reference section 13.2 for spiking protocol.
- Section 14.4.1: indicate volume verification daily for representative vial.
- Section 15.2: Improved fit to make sure no information is truncated.
- Table 2: Analyst Review Checklist updated.

Revision 03, 20120716

- The SOP is an update from Revision 02 dated 20100907.
- Autosampler information updated in sections 9 and 14.
- Example MEE run sequences added to sections 13.4 and 14.6.
- Process for preparing individual ICAL, ICV, CCV and LCS standards added to section 13.
- Redundant references to demonstration of capability removed at 12.2 and 14.6 with update in section 16.1.
- Section 15.2 calculation example updated.
- Table references updated.

Revision 02, 09/07/2010

- The SOP is an update from Revision 01 dated 04/28/09
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory’s revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.
- Calculations have been added.

Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
3. Detection Limit
4. Scope of Application, Including components to be Analyzed
5. Summary of the Test Method
6. Definitions
7. Interferences
8. Safety
9. Equipment & Supplies
10. Reagents and Standards
11. Sample Collection, Preservation, Shipment, and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Data Analysis and Calculations
16. Method Performance
17. Pollution Prevention
18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Contingencies for Handling out-of-control or unacceptable data
20. Waste Management
21. References
22. Tables, Diagrams, Flowcharts and Validation Data

METHANE, ETHANE, ETHENE IN AQUEOUS SAMPLES BY MODIFIED RSK-175 (AUTOMATED HEADSPACE)

1.0 Identification of the Test Method

The GC/FID/Headspace system is used to analyze methane, ethane, and ethene in aqueous samples using method RSK-175.

2.0 Applicable Matrix or Matrices

Aqueous samples

3.0 Detection Limit

Analyte	MDL/DL	LOD	LOQ/RL	Units
Ethane	1.00	2.00	4.00	ug/L
Ethene	1.00	2.00	4.00	ug/L
Methane	1.00	2.00	4.00	ug/L

4.0 Scope of Application, Including Components to Be Analyzed

The GC/FID/Headspace system is used to analyze methane, ethane, and ethene in aqueous samples.

5.0 Summary of the Test Method

This test is used to determine the amount of Methane, Ethene and Ethane in an aqueous sample. Samples are collected using 40mL VOA vials without headspace. Each sample is analyzed using a Headspace autosampler and GC with FID detector.

6.0 Definitions

Laboratory Quality System SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7.0 Interferences

Methane found in the lab environment can be a source of contamination.

8.0 Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.

8.2 Care should be used in handling all samples.

8.2.1 Safety glasses must be worn in the lab at all times. The use of gloves and lab coats is highly recommended.

8.2.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

8.2.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves within the Data Quality Managers Office.

8.2.4 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be

done in a hood. These compounds have been classified as known or suspected human or mammalian carcinogens.

9.0 Equipment & Supplies

- 9.1 Gas Chromatograph:
HP 5890 Series II (temperature programmable).
- 9.2 Autosampler:
HP 7694 Headspace Autosampler
- 9.3 Columns-Capillary columns:
Carboxen 1006 PLOT column–30 meter x 0.53mm ID(or equivalent)
- 9.4 Data Acquisition and Processing Software:
 - 9.4.1 HP Chemstation system is interfaced to the HP-GC for data acquisition, processing, and storing acquired data.
- 9.5 Detector
Flame Ionization Detector
- 9.6 Glassware
 - 9.5.1 25ml Syringe
 - 9.5.2 20ml headspace crimp vials with crimp tops (Restek #24685/#21761 or equivalent)
 - 9.5.3 Gastight syringes - 10 μ L, 100 μ L, 500 μ L and 1mL
 - 9.5.4 5ml dispenser for DI water (calibrated each day used to accurately fill 5ml class A volumetric)

10.0 Reagents and Standards

- 10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory.
- 10.2 Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Certified stock standards are purchased from Restek, Protocol, Ultra and other vendors depending on their availability. The date they are received is noted on the label or container they are received in and in the LIMS system. The date the standards are opened they are recorded and given a sequential number in the LIMS system.
- 10.3 Gas standards are purchased from Restek and other vendors depending on their availability. The date they are received is noted on the container they are received in. The standards are given a sequential number the day they are opened and this is noted on the tank. Standards for MEE are Scotty gases purchased in pressurized tanks at concentration of 1% – Part#23462.

11.0 Sample Collection, Preservation, Shipment, and Storage

Section 3.0 of the EL Quality Assurance Manual include details concerning sample preservation, containers and handling of volatile samples. Samples are collected in 40 ml VOA vials (with 1:1 HCl preservative or without preservative) and shipped to the lab in coolers with ice. Water samples are stored in the water walk-in cooler at a temperature of 0°C-6°C.

12.0 Quality Control

12.1 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.

13.0 Calibration and Standardization

13.1 A minimum five point calibration curve must be introduced into the GC and analyzed for each analyte of interest using the appropriate instrument parameters prior to running samples. If the percent relative standard deviation (% RSD) of the calibration factor is less than 20 percent over the working range, linearity through the origin can be assumed and the average calibration factor can be used in place of a calibration curve. If % RSD is over 20 percent, Linear or Quadratic ranges may be used (linear curve corr. >0.995, quadratic >0.99 with six points). The curve is then verified using a second source standard – separate lot from the calibration standards (80-120% criteria).

13.2 Calibration standards at a minimum of five levels are prepared as follows:

13.2.1 Clean syringes by rinsing with methanol then acetone then pumping with air 3 times to ensure no plugging.

13.2.2 Calibrate the DI dispenser to accurately fill 5ml Class A volumetric,

13.2.3 Add 15ml DI water to a 20ml headspace vial using three pumps of the DI water dispenser set to dispense 5mL.

13.2.4 Cap immediately and check cap for secure fastening.

13.2.5 Turn on Scotty Gas.

13.2.6 Insert appropriately cleaned gas-tight syringe, remove plunger and listen for hissing.

13.2.5.1 If no hiss – clean syringe or change flow meter septum.

13.2.5.2 If hiss, insert plunger to appropriate volume and remove syringe.

13.2.7 Insert syringe at periphery of cap's septum and inject gas through below water's surface watching for bubbles.

13.2.8 Shake all bubbles free and analyze immediately.

13.2.9 Common Calibration/Spike Standards are listed below:

Description	Syringe (μL)	Amount (μL)
Cal1	10	2.5
Cal2	10	5.0
Cal3	10	10
Cal4	100	50
Cal5	100	100
Cal6	1000	500
Cal7	1000	1000
Cal8	1000	2000
ICV	1000	500 (2 nd lot)
CCV	1000	500
BS/MS/MSD	1000	500 (2 nd lot)

- 13.3 The calibration curve must be verified every day samples are run through the analysis of a mid-level standard at the beginning of the sequence, after every 10 field samples and at the end of the sequence. The percent difference back to the curve must not exceed 20 percent. If this requirement is not met, corrective action must be taken before sample analysis continues. Usually this involves recalibration or checking the gastight syringes.
- 13.4 A typical calibration sequence follows
- Instrument blank
 - Calibration Point 1 through Calibration Point 8
 - Initial Calibration Verification
 - Sample analysis could continue as per section 14.6.

14.0 Procedure

The following information describes the instrument and QC requirements to analyze by this method.

14.1 Instrumentation

14.1.1 GC

- a. Initial Temperature: 35 ° C hold for 3.0 minutes.
- b. Ramp: 25 °C/minute to 225 ° C.
- c. Final Temperature 225 ° C hold for 3.08 minutes (or longer).
- d. Detector Temperature 230 ° C.

14.1.2 Headspace Autosampler

- a. Platen: 30 ° C/Platen Equilibration: 0.50 min.
- b. Sample Equilibration: 2.0 min.
- c. Pressurization: 0.50 min/Pressure with 0.25 min Equilibration
- d. Loop Equilibration: 0.20 min.
- e. Injection Time: 0.75 min.
- f. Valve and Line Temp.: 90°C.

- 14.2 Retention Time (RT) Windows - RT criteria set forth in SW-846 method 8000C are used to set retention time windows. New in-house retention time windows are established after major changes to the system (new temperature program, different column phase or diameter). If the established retention time window is less than ± 0.03 minutes, the window defaults to ± 0.03 minutes. Retention times are updated with the first CCV of the day or the mid-level standard of the curve if samples are analyzed in the same sequence as a curve.

- 14.3 **Table 1** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

14.3.1 A method blank is required every 20 samples. Any detected concentration should not exceed $\frac{1}{2}$ the project reporting limit.

14.3.2 A Laboratory Control Sample (LCS) or Blank Spike (BS) is required every 20 samples. An LCS Duplicate (LCSD) or BS Duplicate (BSD) is used if sample volume is insufficient for running MS/MSD. See section 13.2 for spiking instructions. Limits 80-120 %.

14.3.3 An MS/MSD pair is run (if sufficient volume is received) upon client request every 20 samples per matrix. See section 13.2 for spiking instructions. Limits

80-120 %.(If insufficient volume is received, an LCSD/BSD may be used to demonstrate precision.)

14.4 Sample preparation includes the following steps:

- 14.4.1 Verify the volume of a representative crimp-top vial from the lot being used by filling with water and measuring to the nearest decimal place with the 25ml syringe. Subtract 15ml then record the result in the LIMS bench sheet as headspace volume.
- 14.4.2 Rinse the 25ml gas-tight syringe with DI water.
- 14.4.3 Remove the plunger and invert with gloved finger over the tip.
- 14.4.4 Fill syringe to greater than 15mL with sample and replace plunger.
- 14.4.5 Invert syringe and displace any air bubbles.
- 14.4.6 Measure 15ml and add to headspace vial.
- 14.4.7 Cap immediately and check for secure fastening.
- 14.4.8 Load onto the autosampler along with a similarly prepared method blank containing 15 ml of DI water, a blank spike/blank spike duplicate (BS/BSD, BSD only if no Matrix Spike/Matrix Spike Duplicate or MS/MSD volume).
- 14.4.9 A mid-level standard (continuing calibration verification or CCV) must be run at the beginning and end of the sequence and after every 10 field samples within the sequence. The mid-level standard cannot exceed 20 percent difference from the initial calibration.
- 14.4.10 A typical sample sequence follows:
 - Instrument Blank
 - CCV 1
 - Method Blank
 - Blank Spike
 - Blank Spike Duplicate (NA if MS/MSD volume available)
 - Field Sample 1 – Field Sample 10
 - Matrix Spike
 - Matrix Spike Duplicate
 - CCV 2
 - Field Sample 11 – Field Sample 20
 - CCV 3

14.5 Following sample analysis, the data is reduced using the Chemstation data system. The retention times are updated with the first midpoint check of the day or from the midpoint of the calibration curve if analyzed before the samples. The following must be checked to see if the samples will require re-analyses or dilution.

- 14.5.1 The analyte concentration/area count must be within the range of the calibration curve. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover. Any dilution should keep the concentration of the analyte in question within the top half of the curve.

15.0 Data Analysis and Calculations

- 15.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

15.2 Calculations:

$$\text{Calibration Factor (CF)} = \frac{\text{Response}}{\text{Dec Equiv} * 1000}$$

	Column B	Column C
4	Sample	Example
6	Methane Instrument Reading (DE*1000)	0
10	Ethene Instrument Reading (DE*1000)	0
11	Ethane Instrument Reading (DE*1000)	0
13	Methane (µg/L)	=C29
15	Ethene (µg/L)	=C40
16	Ethane (µg/L)	=C51
20	Samples	Methane
21	DE Calculation (Instrument Reading/1000)	=C6/1000
22	$x(g) = \text{DE}/\text{Henry's Law Constant}$	=C21/44900
23	$n(g) = 55.5 \text{ (g-mole for 1L water)} * x(g) * 1\text{ATM}/(1\text{ATM}+2\text{PSI})$	=55.5*C22/1.137
24	$C \text{ (mg cpd/L water)} = n(g)*\text{MW}*(1000\text{mg/g})$	=C23*16000
25	$D \text{ (g/L)} = \text{MW}/(22.4\text{L/mole})(\text{sample temp } ^\circ\text{K}/273^\circ\text{K})$	=16/(22.4*(303/273))
26	$A(h) \text{ mL cpd} = (\text{mL headspace})*\text{DE}$	=6.2*C21
27	$A(l) \text{ (mg cpd/L water)} = A(h)/(\text{Sample Volume in L})*(D)(1000\text{mg/g})(1\text{L}/1000\text{mL})$	=(C26/0.015)*(C25)
28	$\text{TC (mg cpd/L water)} = A(l) + C$	=C27+C24
29	$\text{TC (}\mu\text{g cpd/L water)} = \text{TC mg/L} * (1000\mu\text{g}/1\text{mg})$	=C28*1000
31	Samples	Ethene
32	DE Calculation (Instrument Reading/1000)	=C10/1000
33	$x(g) = \text{DE}/\text{Henry's Law Constant}$	=C32/12700
34	$n(g) = 55.5 \text{ (g-mole for 1L water)} * x(g) * 1\text{ATM}/(1\text{ATM}+2\text{PSI})$	=55.5*C33/1.137
35	$C \text{ (mg cpd/L water)} = n(g)*\text{MW}*(1000\text{mg/g})$	=C34*28000
36	$D \text{ (g/L)} = \text{MW}/(22.4\text{L/mole})(\text{sample temp } ^\circ\text{K}/273^\circ\text{K})$	=28/(22.4*(303/273))
37	$A(h) \text{ mL cpd} = (\text{mL headspace})*\text{DE}$	=5.167*C32
38	$A(l) \text{ (mg cpd/L water)} = A(h)/(\text{Sample Volume in L})*(D)(1000\text{mg/g})(1\text{L}/1000\text{mL})$	=(C37/0.015)*(C36)
39	$\text{TC (mg cpd/L water)} = A(l) + C$	=C38+C35
40	$\text{TC (}\mu\text{g cpd/L water)} = \text{TC mg/L} * (1000\mu\text{g}/1\text{mg})$	=C39*1000
42	Samples	Ethane
43	DE Calculation (Instrument Reading/1000)	=C11/1000
44	$x(g) = \text{DE}/\text{Henry's Law Constant}$	=C43/34200
45	$n(g) = 55.5 \text{ (g-mole for 1L water)} * x(g) * 1\text{ATM}/(1\text{ATM}+2\text{PSI})$	=55.5*C44/1.137
46	$C \text{ (mg cpd/L water)} = n(g)*\text{MW}*(1000\text{mg/g})$	=C45*30000
47	$D \text{ (g/L)} = \text{MW}/(22.4\text{L/mole})(\text{sample temp } ^\circ\text{K}/273^\circ\text{K})$	=30/(22.4*(303/273))
48	$A(h) \text{ mL cpd} = (\text{mL headspace})*\text{DE}$	=5.167*C43
49	$A(l) \text{ (mg cpd/L water)} = A(h)/(\text{Sample Volume in L})*(D)(1000\text{mg/g})(1\text{L}/1000\text{mL})$	=(C48/0.015)*(C47)
50	$\text{TC (mg cpd/L water)} = A(l) + C$	=C49+C46
51	$\text{TC (}\mu\text{g cpd/L water)} = \text{TC mg/L} * (1000\mu\text{g}/1\text{mg})$	=C50*1000
62	A(h) = milliliters of analyte in the headspace	MW = molecular weight of the gas
63	A(l) = analyte in liquid phase	n(g) = moles of gas
64	C = saturation concentration of the gas	ST = sample temperature
65	D = density	TC = total concentration of analyte in the original sample
66	DE = decimal equivalents for the partial pressure of the gas	x(g) = equilibrium mole fraction of the dissolved gas
67		
68	Element AnalyteInfo1 {ALYTEINFO1} = Density (0.64356M,1.1262Ee,1.2067Ea)	
69	Element AnalyteInfo2 {ALYTEINFO2} =Henry's Law Constant (44900M,12700Ee,30200Ea)	
70	Element AnalyteInfo3 {ALYTEINFO3} =MW*1000 (16000M,28000Ee,30000Ea)	
71	Concentration = (((6.2* {IRESULT}) /0.015* {ALYTEINFO1})+(({IRESULT} / {ALYTEINFO2})*55.5/1.137* {ALYTEINFO3}))*(DILN)	

16.0 Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-BS samples spiked at approximately 4x the LOQ. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality Assurance Manual is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 1** for acceptance criteria.

17.0 Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 1** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 1** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20.0 Waste Management

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.0 References

- 21.1 Newell, Bryan, RSKSOP-175, Rev.0, August 1994.
- 21.2 Newell, Bryan, RSKSOP-147, Rev.0, January 1993.
- 21.3 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Methods 8000C and 8015C.

22.0 Tables, Diagrams, Flowcharts and Validation Data

- 22.1 Table 1, quality control criteria
- 22.2 Table 2, analyst review checklist

Table 1. Organic Analysis by Gas Chromatography (Method RSK-175)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f of Dod QSM 4.1).	Not Applicable (NA).	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL determination	Initial method demonstration required for some states – not required for DoD	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis then quarterly verification.	Detected at the LOD concentration.			
LOQ establishment and verification	Prior to initial analysis then quarterly verification.	Recovered within LCS (BS) limits at 1-4x the LOQ concentration.			
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study or 0.03min., whichever is greater.	NA.	NA.	
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	One of the options below: Option 1: RSD for each analyte $\leq 20\%$ Option 2: linear least squares regression: $r \geq 0.995$ or $r^2 \geq 0.990$ Option 3: non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin for DoD projects.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte	Once per ICAL and at the beginning of the analytical shift.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. All project analytes within $\pm 20\%$ of expected value from the ICAL	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples should be run until calibration has been verified.
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples (maximum of 20 for non-DoD projects) , and at the end of the analysis sequence.	All project analytes within established retention time windows. All project analytes within $\pm 20\%$ of expected value from the ICAL	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results should not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected $> \frac{1}{2}$ RL and $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct problem, If required, prep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory control sample (LCS or BS) containing all analytes to be reported, including surrogates	One per preparatory batch.	All project analytes within $\pm 20\%$ of expected value	Correct problem, then prep and reanalyze the LCS (BS) and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS (BS). Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table 1. Organic Analysis by Gas Chromatography (Method RSK-175)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix.	For matrix evaluation, use LCS (BS) acceptance criteria above.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS (BS) limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix.	MSD: For matrix evaluation, use LCS (BS) acceptance criteria above. MSD or sample duplicate: $RPD \leq 30\%$ (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Results reported between DL and LOQ	NA	NA	NA	Apply J-flag to all results between DL and LOQ.	

Table 2.
ANALYST DATA REVIEW CHECKLIST

Sample Number(s):				
Batch Number(s):				
Sequence Number:		Run Date:	Instrument ID:	
Method:	RSK-175	Calibration:	NCR:	

QA/QC Item	Yes	No	NA	2nd Review
A. Initial Calibration				
1. Does the curve consist of at least five Calibration Standards (six for quadratic curve)?				
2. Is the low standard equal to or below the LOQ?				
3. Are the %RSD or fit criteria within QC limits for all analytes?				
B. Second Source Verification				
1. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?				
C. Continuing Calibration				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 10 samples (20 non-DoD) or every 12 hours and at the end of the sequence?				
2. Are the % differences within QC limits for all analytes?				
D. Sample Analysis				
1. Are all sample holding times met?				
2. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?				
E. QC Samples				
1. Is the Method Blank extracted at the desired frequency and is its concentration for target analytes less than the LOD?				
2. Is the Laboratory Control Sample/Blanks Spike extracted at the desired frequency and are its percent recoveries within QC limits?				
3. Is the Matrix Spike/Matrix Spike Duplicate extracted at the desired frequency and are the percent recoveries/RPDs within QC limits?				
F. Others				
1. Are all nonconformances included and noted?				
2. Are all calculations checked at the minimum frequency with one example worked on the raw data?				
3. Did analyst initial/date the appropriate printouts and report sheets?				
4. Are all manual integrations checked by a second reviewer to verify they were performed correctly?				
F. LIMS				
1. Data uploaded to LIMS correctly with associated primary analyst correct?				
2. Sample _____ showing full re-calculation from raw data through to final concentration in LIMS.				
Comments on any "No" response:				

Analyst: _____

Date: _____

Second-Level Review: _____

Date: _____

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP221 REVISION #: 15 EFFECTIVE DATE: 20131011

TOTAL ORGANIC CARBON (TOC)
By SM 5310 C-2011, SW846 METHOD 9060/9060A or
Lloyd Kahn Method "Determination of TOC in Sediment"

APPROVALS:

Lab Director:

Date: 10/11/2013

Data Quality Manager:

Date: 10/11/2013

Section Supervisor:

Date: 10/11/2013

Changes Summary

Revision 15, 10/11/2013

- Added water system software TOC-Vws/TOC-Control V version 1.07.00.00sp1 and soil system software WinTOC3.0.

Revision 14, 08/13/2013

- References to quarterly calibration removed – no method criteria identified.

Revision 13, 07/15/2013

- Procedure updated to remove single analysis option on 9060/9060A method water analysis.

Revision 12, 07/01/2013

- Standard method reference updated to reflect “-2011” and SOP verified to include necessary QC.
- Added demonstration of capability requirement to section 12.
- Clarified 5310C limits for CCVs.
- Underlined section 8.7 indication that strict 9060 samples are analyzed in quadruplicate.

Revision 11, 09/12/2012

- Standard method reference format updated and QS references updated
- QC frequencies and criteria updated for SM 5310 C.
- Duplicate/Quadruplicate analysis requirements clarified.
- 6.3 - standard makeup updated.
- 6.7 - solution stabilization added.
- 8.2 - instrument warmup added.
- 8.3 and 9.6 - sample sequence updated.
- 9.11 - added instructions for printing the run log
- 10.7 - added MRL standard analysis.
- Listing of attachments added as section XVI with data review checklist added as Attachment 2.

Revision 10, 08/22/2011

- The SOP is an update from Revision 09 dated 07/12/2010
- Section 8.9 added “When verifying the sample calculations, note the instrument manual (p46) indicates **“The calibration curve is shifted through the origin to correct for the TC contained in purified water used for preparation of the standard solutions.”** Therefore, the sample concentration is calculated by dividing the instrument response by the curve slope and ignoring the intercept.”

Revision 09, 07/12/10

- The SOP is an update from Revision 08 dated 04/28/09
- The SOP has been reviewed and confirmed to be accurate.
- The soil TOC method calibration concentrations were updated.
- The LCS reference was changed to BS.

Table of Contents

- I. SCOPE AND APPLICATION
- II. SUMMARY OF METHOD
- III. SAMPLING HANDLING AND PRESERVATION
- IV. INTERFERENCES
- VI. REAGENTS/STANDARDS
- VII. INSTRUMENTATION
- VIII. AQUEOUS SAMPLE PROCEDURE
- IX. SOIL/SEDIMENT SAMPLE PROCEDURE
- X. CORRECTIVE ACTIONS
- XII. HEALTH AND SAFETY
- XIII. WASTE DISPOSAL and POLLUTION PREVENTION
- XIV. METHOD PERFORMANCE
- XV. REFERENCES
- XVI. ATTACHMENTS
 - 1. Appendix I – Basic TOC start-up notes for analysis.
 - 2. Data Review Checklist

TOTAL ORGANIC CARBON (TOC)
BY SM 5310 C-2011, SW846 METHOD 9060/9060A or
Lloyd Kahn Method "Determination of TOC in Sediment"

I. SCOPE AND APPLICATION

SM5310C is used to determine the concentration of organic carbon in source and drinking water, SW846 Method 9060/9060A is used to determine concentrations of carbon in saline waters, domestic and industrial wastes and can be modified for soil determination. The Lloyd Kahn Method is used for determination of TOC in solid matrices. These methods should be read over carefully by the analyst and any restrictions should be noted.

II. SUMMARY OF METHOD

The organic carbon is measured using an Shimadzu Total Organic Carbon Analyzer (aqueous samples) and an OI Analytical Solids TOC Analyzer model 1010 (soil/sediment samples). The Shimadzu instrument converts the organic carbon in a sample using wet chemical oxidation. The CO₂ formed is then measured by an infrared detector (replaces ultraviolet detector in SM 5310C). With the model 1010 Solids TOC analyzer, TOC is determined by acidifying a sample and heating it to 250°C to remove the TIC. The sample is then heated to 900°C to combust the remaining TOC. The resulting carbon dioxide from the TOC is detected by a non-disperse infrared (NDIR) detector that has been calibrated to directly display the mass of carbon dioxide detected. This mass is proportional to the mass of TOC in the sample.

S/W	Analyte	MRL/LOQ	LOD	MDL/DL	Units
Water	Total Organic Carbon	3.0	2.50	1.25	mg/L
Solid	Total Organic Carbon	800	400	200	mg/Kg

V. SAMPLING HANDLING AND PRESERVATION

Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

- 3.1 Sampling and storage in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples. NOTE 1: A brief study performed in the EPA Laboratory indicated that distilled water stored in new, one quart cubitainers did not show any increase in organic carbon after two weeks exposure.
- 3.2 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the lapse of time between collection of samples and start of analysis should be kept to a minimum. The holding time is 28 days for waters and soils with the exception of the Lloyd Kahn method soils, which requires a 14 day holding time. Also, samples must be kept cool (4°C) and protected from sunlight and atmospheric oxygen.
- 3.3 When water samples cannot be analyzed immediately, the sample is preserved by acidification to (pH <= 2) with HCl or H₂SO₄. Both water and soil samples are stored at 4°C.

VI. INTERFERENCES

4.1 WATER METHOD

- 4.1.1 Removal of carbonate and bicarbonate carbon by acidification and purging with purified gas results in the loss of volatile organic substances. The volatiles also can be lost during sample blending, particularly if the temperature is allowed to rise. Another important loss can occur if large carbon-containing particles fail to enter the needle used for injection. Filtration although necessary to eliminate particulate organic matter when only DOC is to

be determined, can result in loss or gain of DOC, depending on the physical properties of the carbon-containing compounds and the adsorption of carbonaceous material on the filter, or its desorption from it. Check filters for their contribution to DOC by analyzing a filtered blank. Note that any contact with organic material may contaminate a sample. Avoid contaminated glassware, plastic containers, and rubber tubing. Analyze treatment, system, and reagent blanks.

This procedure is applicable only to homogenous samples which can be injected into the apparatus reproducibly by means of a pipette. The openings of the pipette limit the maximum size of particles which may be included in the sample.

4.2 SOIL METHOD

- 4.2.1 All materials must be routinely demonstrated to be interference –free under the analysis conditions by running blanks. Use high purity or purified reagents and gases to help minimize interference problems.
- 4.2.2 The infrared detector is sensitized to CO₂ and accomplishes virtually complete rejection of response from other gases that absorb energy in the infrared region.

V. DEFINITIONS

Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

- 5.1 ANALYTICAL BATCH-The set of samples extracted /distilled/ or digested at the same time to a maximum of 20 samples.
- 5.2 CALIBRATION BLANK (CB)- A volume of reagent water in the same matrix as the calibration standards, but without the analyte.
- 5.3 CALIBRATION STANDARD (CAL)- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
- 5.4 FIELD BLANK (FB)- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, including exposure to a sample bottle holding time, preservatives, and all preanalysis treatments. The purpose is to determine if the field or sample transporting procedures and environments have contaminated the sample.
- 5.5 FIELD DUPLICATE (FD)- Two samples taken at the same time and place under identical circumstances which are treated identically throughout field and laboratory procedures. Analysis of field duplicates indicates the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.
- 5.6 LABORATORY BLANK (BLK)- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, except that it is not taken to the sampling site. The purpose is to determine if the analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.
- 5.7 LABORATORY CONTROL SAMPLE (BS)- A solution prepared in the laboratory by dissolving a known amount of one or more pure compounds in a known amount of reagent water. Its purpose is to assure that the results produced by the laboratory remain within the acceptable limits for precision and accuracy. (This should not be confused with a calibrating standard, it must be prepared from a source other than the same source as the calibration standards).
- 5.8 LABORATORY DUPLICATE (DUP)- Two aliquots of the same environmental sample treated identically throughout a laboratory analytical procedure. Analysis of laboratory duplicates indicates precision associated with laboratory procedures but not with sample collection, preservation, or storage procedures.
- 5.9 QUALITY CONTROL CHECK SAMPLE, also known as INITIAL CALIBRATION VERIFICATION (ICV)- A sample containing analytes of interest at known concentrations (true

value) of analytes. The ICV is obtained from a source external to the laboratory or is prepared from standards obtained from a different source than the calibration standards. The purpose is to check laboratory performance using test materials that have been prepared independently from the normal preparation process.

5.10 MINIMUM REPORTING LIMIT (MRL) – the lowest standard concentration to be reported. Supported by the daily analysis of a standard at the specified concentration.

5.11 METHOD DETECTION LIMIT (MDL)- The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero. Determined annually.

VII. REAGENTS/STANDARDS

The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. All certificates received with standards must be marked with the LIMS ID and forwarded to the administration department for scanning/saving in order to be available for view within LIMS. All reagents used for preparation and analysis are entered into LIMS. These reagents are added to the batch sheet when the samples are batched to ensure traceability of the reagents used to the associated samples. All reagents shall be made from ACS reagent grade chemicals.

6.1 The laboratory reagent blank water used for TOC analysis is obtained from the Modulab Analytical water purification system in the analytical laboratory. **Boiling the water is not performed as the method requests. This has not presented a problem.**

6.2 Potassium hydrogen phthalate, primary stock solution, 1000 mg/L: Dissolve 0.2128g of potassium hydrogen phthalate (primary standard grade) in 100.0 mL water.

6.3. Potassium hydrogen phthalate, standard solutions : A 25 mg/L standard is prepared by diluting 5 mLs of the 1000 mg/L Primary stock solution to 200 mLs. Also, a 50 mg/L std. is prepared by diluting 10 mLs of the 1000 mg/L Primary stock solution to 200 mLs. These two standards are analyzed alternately during the analytical run.

6.4. The carbonate-bicarbonate solutions are not needed for this instrument.

6.5 Calibration Standards

1. For the water method, calibration standard is Potassium Hydrogen Phthalate. Standards are made from dilutions of the stock 1000 mg/L standard as follows:

1.0 mg/L = 0.10 mL of 1000 mg/L -> 100 mL

2.5 mg/L = 0.25 mL of 1000 mg/L -> 100 mL

5.0 mg/L = 0.50 mL of 1000 mg/L -> 100 mL

10.0 mg/L = 1.0 mL of 1000 mg/L -> 100 mL

25.0 mg/L = 5.0 mL of 1000 mg/L -> 200 mL

50.0 mg/L = 10.0 mL of 1000 mg/L -> 200 mL

100 mg/L = 10.0 mL of 1000 mg/L -> 100 mL

2. A low level standard curve must be run for drinking water samples with the standards made as follows:

0.25 mg/L = 0.025 mL of 1000 mg/L -> 100 mL

0.50 mg/L = 0.050 mL of 1000 mg/L -> 100 mL

1.0 mg/L = 0.10 mL of 1000 mg/L -> 100 mL

1.5 mg/L = 0.15 mL of 1000 mg/L -> 100 mL

2.5 mg/L = 0.25 mL of 1000 mg/L -> 100 mL

5.0 mg/L = 0.50 mL of 1000 mg/L -> 100 mL

10.0 mg/L = 1.0 mL of 1000 mg/L -> 100 mL

3. The soil method calibration standard is prepared by using an OI commercially prepared 30% carbon sucrose solution.

7.6 Laboratory Control Sample/Blank Spike (BS):

1. For the water method, the BS is normally made from a performance evaluation solution of which the true value is known. This solution is given a unique identifier.
2. For the soil method, the BS is made from a 30% sucrose solution which is made by weighing up 7.125 grams of EM Reagent Grade Sucrose and diluting to 10 mL with deionized water volumetrically.
- 6.7. Persulfate oxidation solution: This solution is made by dissolving 60g of sodium persulfate in DI water, adding 15 ml of phosphoric acid and diluting to 500 ml. Allow solution to stabilize 24 hours.
- 6.8. Phosphoric acid solution: Dilute 100 mL of concentrated 85% phosphoric acid in 500 mL of water. This is used for water.
- 6.9. Phosphoric acid solution 5%: Dilute 59 mL of concentrated 85% phosphoric acid in 1000 mL of water. This is used for soil.

VII. INSTRUMENTATION

- 7.1 The instrument used for the Water TOC analysis is a Shimadzu Total Carbon Analyzer with TOC-Vsw/TOC-Control V version 1.07.00.00sp1 software. An OIC 1010 soil/sediment carbon analyzer is used for soil samples with WinTOC3.0 software.
- 7.2 There is a Shimadzu autosampler which will hold 68 samples.
- 7.3 The corresponding data for each sample is obtained from the Shimadzu software for the water samples. The soil/sediment data are printed out at the organic GC printer.

VIII. AQUEOUS SAMPLE PROCEDURE

- 8.1 Wearing labcoat, gloves and safety glasses, the standards and check solutions should be taken out of the refrigerator and allowed to warm to room temperature. Also, remove samples from sample storage signing them out appropriately on the internal chain of custody form. Fresh acid and oxidation solutions should be poured into the appropriate containers on the front of the instrument.
- 8.2 Follow the instructions for operation of the instrument in Chapter 4, section 4.3 of the Shimadzu Model TOC-VWS User Manual. **See Appendix I. for Basic TOC start-up notes for analysis.**

NOTE: Instrument needs to warm up at least 30 minutes before analysis begins.

- 8.3 Following is a list outlining the order in which the samples should be analyzed. Each sample VOA vial should be numbered and its identity entered into the TOC schedule. Note: All blanks should be acidified to pH 2 to match the matrix of the samples analyzed. Analysis may begin with CCV/CCB/MRL if an ICAL is not analyzed.
 1. 100 ppm
 2. 50 ppm
 3. 25 ppm
 4. 10 ppm in duplicate
 5. 5.0 ppm in duplicate
 6. 2.5 ppm in duplicate
 7. 1.0 ppm in duplicate
 8. Method blank in duplicate
 9. Blank spike (BS/ICV) in duplicate
 10. Up to 10 field samples (+ QC) in duplicate
 11. 25 ppm CCV in duplicate
 12. Method blank in duplicate (if 5310C, every 10 samples)
 13. Blank spike (BS/ICV) in duplicate (if 5310C, every 10 samples)
 14. Up to 10 field samples (+ QC) in duplicate
 15. 50 ppm CCV in duplicate

NOTE – Duplicate analyses are automatically repeated if sample results are not within $\pm 10\%$. Aqueous samples analyzed by 9060/9060A must be analyzed in quadruplicate.

- 8.8 Instrument printouts are generated from the software. Normal procedure is followed for preparing reports and the data is second checked before being given to the supervisor.
- 8.9 When verifying the sample calculations, note the instrument manual (p46) indicates "**The calibration curve is shifted through the origin to correct for the TC contained in purified water used for preparation of the standard solutions.**" Therefore, the sample concentration is calculated by dividing the instrument response by the curve slope and ignoring the intercept.

XI. SOIL/SEDIMENT SAMPLE PROCEDURE

A sample is introduced into the Solid Module via a conditioned sample cup. Once the sample has been introduced the entire analysis sequence is automatic. Please reference Chapter 4 of the OI 1010 Solid Module instrument manual for instrument states and configuration when initially setting the instrument methods up.

TC Mode Instrument Settings:

- Analysis Temp: 900°C
- Analysis Time: 6.5 minutes
- Nitrogen Gas Flow: 60-100 psi (external regulator regulator)
- Oxygen Gas Flow: 40-60 psi (external regulator)

Following is a step by step description of a routine soil TOC analysis.

- 11.1 The standards and check solutions should be taken out of the refrigerator and allowed to warm to room temperature. The nitrogen and oxygen (internal regulator should be set at 50-60 psi) turned on allowing a nitrogen flow of 350-400 mL/minute and an oxygen flow of 180 mL/minute (± 3 mL/minute).

NOTE: DO NOT TURN THE ANALYZER ON BEFORE TURNING THE GAS ON!

- 11.2 Let the gas flow through the instrument for a few minutes. The instrument should now be turned on and allowed to stabilize for 30 minutes.
- 11.3 Condition the cups (with quartz wool in them) using Diagnostics under Instrument Menu commands, (don't condition too many cups at a time since sitting in contact with the air can cause contamination).
- 11.4 Set up the subdirectory (using the current date to ID it) under WinTOC output.
- 11.5 If doing an initial calibration curve use an appropriate μL syringe to make the following measurements of the sucrose standard in order to achieve the indicated concentrations. Make sure that there are no air bubbles in the syringe. Turn the syringe with the needle pointed up and vibrate the barrel and disperse any air from the syringe. To enter the calibration information on the instrument go to Instrument Cal Menu, type in the calibration standard values and save the file as the cal.. date analyzed.

μL 30% Sucrose STD	Concentration (mg)
0	0
2.0 (1:6 solution)	0.10
3.0	0.90
25	7.5
50	15

Note: The 1:6 solution of the 30% Sucrose standard is prepared by mixing 100 μL of the 30% Sucrose standard with 500 μL of water.

- 11.6 Analysis may begin with CCV/CCB/MRL if an ICAL is not analyzed. Enter the sequence to be analyzed as listed below:

1. CCV(CCV1+ date analyzed for ID) or Initial calibration – single analyses
2. CCB1 (Or ICB)
3. MRL standard
4. Method Blank(MB + date analyzed for ID) – single analyses
5. BS, 15 mg dextrose (BS + date analyzed for ID) – single analyses
6. Up to 10 field samples (+ QC) in duplicate
7. CCV(CCV2+ date analyzed for ID) – single analyses
8. CCB2 (CCB2+ date analyzed for ID) – single analyses
9. Up to 9 field samples (+ QC) in duplicate
10. One field sample in quadruplicate
11. CCV (CCV3+ date analyzed for ID) – single analyses
12. CCB (CCB3+ date analyzed for ID) – single analyses
13. Up to 10 field samples (+ QC) in quadruplicate
14. CCV(CCV4+ date analyzed for ID) – single analyses
15. CCB (CCB4+ date analyzed for ID) – single analyses
16. Up to 9 field samples (+ QC) in duplicate
17. One field sample in quadruplicate
18. FCV(CCV5+ date analyzed for ID) – single analyses
19. FCB(CCB5+ date analyzed for ID) – single analyses

NOTE – soils analyzed by modified 9060/9060A are analyzed in quadruplicate.

- 11.7 Samples should be stored away from light and at 4°C. Wearing labcoat, gloves and safety glasses remove samples from sample storage signing them out appropriately on the internal chain of custody form.
- 11.8 Transfer a homogeneous aliquot(~5 g) of the sample into a small pre-labeled aluminum weighing pan. Label each pan with the appropriate sample ID then add enough phosphoric acid (1-2 ml) to remove the Total inorganic carbon (TIC) when the sample is placed in an oven at 250°C. Place the samples in the 250°C oven for 10 minute and begin prepping the sample cups to weigh 0.2g-1.0g of each sample (in duplicate or quadruplicate, as required). Limit the time that the cups are exposed to the atmosphere as to reduce potential contamination. **Note: Since the samples are dried in this manner before the sample aliquot it taken, a % solids determination and calculation is NOT necessary to report the sample concentrations in dry weight.** After samples are dried, crush samples using a clean mortar and pestle.
- 11.9 Set the OI 1010 to the TC Mode and start running the sequence beginning with the initial calibration or calibration verification standard as illustrated above. Weigh each sample in duplicate or quadruplicate, making sure to limit the time that samples are exposed to the atmosphere.
- 11.10 The Excel file for calculations is located in “V:\WCM\TESTS\TOC soil\”. The sample identity, its corresponding mgC reading, and the sample weight are entered into the appropriate columns. The Excel worksheet is self explanatory. Normal procedure is followed for preparing reports and the data is second checked before being given to the supervisor.
- 11.11 To print the run log that contains the results, open the Utilities tab in the operating software. Select view Run Log, then select the file that contains the results. The file can then be printed from the window that is opened.

XII. QC REQUIREMENTS

Quality Systems SOP QS08 “Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.

NOTE – Demonstration of Capability (DOC) is required for each analyst prior to analyzing any samples. DOC consists of preparation/analysis of 4 BS samples with recoveries within BS limits for applicable method (or most stringent limits if applying to multiple methods) and RPD within duplicate limit of 20.

- 10.1 The ICAL correlation coefficient requirement is less than 0.995 for r (0.990 for r² in LIMS).
- 10.2 Analyze a laboratory control sample (BS) for each batch of samples **(maximum of 20 soil/water samples per batch except 5310C is maximum of 10 water samples per batch)**. If the BS does not fall within the control limits of 80 to 120%, corrective action must be taken to find and correct the problem. **(5310C limits are 85%-115%)**.
- 10.3 Run a method blank (BLK) for each batch of samples **(maximum of 20 soil/water samples per batch except 5310C is maximum of 10 water samples per batch)**. The BLK should be less than 1/2 the reporting limit or LOQ.
- 10.4 One matrix spike and matrix spike duplicate must be run per set of 20 samples. For water analysis, a spike and spike duplicate are made by mixing 20 mLs of sample with 0.30 mLs of stock 1000 mg/L standard using an ependorf pipette. The true value is 15 mg/L. The percent recoveries on a MS and a MSD should be within 75 and 125%. Relative percent difference (RPD) on duplicates should be less than 20%. If not, a non-conformance report (NCR) must be generated and approved by your supervisor. Note – sample duplicate may be analyzed in place of MSD.
- 10.5 Analyze an initial calibration verification (ICV) immediately after the calibration curve. Analyze a calibration check verification (CCV) standard at the beginning of the sequence, after every tenth field sample and at the end of the sequence. The percent recoveries should be in the range of 80 to 120%. **(5310C limits are 90%-110%)**.
- 10.6 When analyzing water samples, all water blanks before samples and standards must be below the detection limit, otherwise the samples must be rerun.
- 10.7 Analyze an initial calibration blank (ICB) following the ICV. Analyze a continuing calibration blank (CCB) following each CCV. The ICB and CCB should be less than \pm the MDL.
- 10.8 Analyze a MRL standard at the beginning of each sequence – it must recover in the range of 80 to 120%.
- 10.9 Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.

Calculate spikes as follows where everything is in concentration.

$$\% \text{ Recovery} = \frac{\text{Spike} - \text{Sample}}{\text{True Value}} \times 100$$

Relative percent difference is calculated as follows, with everything in concentration:

$$\text{RPD} = \frac{\text{Higher Concentration} - \text{Lower Concentration}}{\text{Average of Concentrations}} \times 100$$

- 10.10 **For aqueous samples check an acidified 20mg/L inorganic carbon standard quarterly, to assure that purge gas flow is adequate to remove inorganic carbon. The result should be below the reported quantitation limit.**

XIII. CORRECTIVE ACTIONS

Quality Control SOP QS05, “Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results”, provides details on handling out of control data.

11.1 INSTRUMENT RELATED

1. ICV not within $\pm 20\%$ or $\pm 10\%$ (5310C)
 - a. If the problem is with the solution.
 - i. Re-prepare, obtain new stock if necessary.
 - b. If the problem is with the calibration.
 - i. Recalibrate through analysis of appropriate standards and recheck ICV.
2. CCV not within $\pm 30\%$ (Soil) or $\pm 20\%$ (Water) or $\pm 10\%$ (5310C)
 - a. If the problem is with the solution.
 - i. Re-prepare, obtain new stock if necessary.
 - b. If the problem is with the calibration.
 - i. Recalibrate through analysis of appropriate standards and re-prepare /reanalyze the previous ten sample according the following guidelines.
 - a. If the CCV was biased high, any of the previous ten samples which were below the minimum detection limit do not require reanalysis.
 - b. If the CCV was biased low, the previous ten samples must be reanalyzed.

*** Incorrectly set gas flow is a common instrument related problem which requires corrective action. Verify that all gas flows are adjusted properly.**

11.2 SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within $\pm 20\%$ aqueous or $\pm 50\%$ soil/sediment
 - i. The associated sample data must be qualified on the final report.
2. Spike analysis recovery not within $\pm 25\%$ aqueous or $\pm 50\%$ soil/sediment
 - i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.
 - ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. A non-conformance report must accompany the data and be emailed or given to the supervisor.

XII. HEALTH AND SAFETY

- 12.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.
- 12.2 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of latex gloves and lab coats is highly recommended.
- 12.3 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
- 12.4 MSDS sheets are available for all reagents and standards that have been purchased. These are located on the bookshelves in the Data Quality Manager's office.

XIII. WASTE DISPOSAL and POLLUTION PREVENTION

- 13.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.
- 13.2 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

XV. METHOD PERFORMANCE

14.1 Precision and Bias for Total Organic Carbon (TOC) by Persulfate-Ultraviolet Oxidation. (Water samples)

Characteristic Of Analysis Concentration determined, mg/L:	Spring Water	Spring Water +0.15 mg/L KHP*	Tap Water	Tap Water +10 mg/L KHP*	Municipal Wastewater Effluent
Replicate 1	0.402	0.559	2.47	11.70	5.88
Replicate 2	0.336	0.491	2.49	11.53	5.31
Replicate 3	0.340	0.505	2.47	11.70	5.21
Replicate 4	0.341	0.523	2.47	11.64	5.17
Replicate 5	0.355	0.542	2.46	11.55	5.10
Replicate 6	0.366	0.546	2.46	11.68	5.33
Replicate 7	0.361	0.548	2.42	11.55	5.35
Mean, mg/L	0.35	0.53	2.46	11.53	5.32
Std. Deviation: mg/L	0.02	0.03	0.02	0.21	0.23
%	6	6	1	2	4
Actual Value, mg/L	-	0.50	-	12.46	-
Recovery, %	-	106	-	93	-
Error, %	-	6	-	7	-

*KHP = potassium hydrogen phthalate.

14.2 There was no method performance data available for the soil procedure.

XV. REFERENCES

1. Annual Book of ASTM Standards, Part 31, "Water," Standard D 2574-79, p. 469 (1976).
2. Standard Methods for the Examination of Water and Wastewater, 21st ED., Method 5310C (2000).
3. EPA SW-846, Method 9060/9060A.
4. Lloyd Kahn Method, "Determination of Total Organic Carbon in Sediment"

XVI. ATTACHMENTS

1. Appendix I – Basic TOC start-up notes for analysis.
2. Data Review Checklist

APPENDIX I. Basic TOC start-up notes for analysis.

1. Power up the lamp for warm –up, check reagents inside instrument cavity to make sure all are filled before starting the run.
2. Fill Fresh DI water in 1 gallon jug; DI squirt bottle and 1 L plastic
3. Label and load VOA vials with standards and samples into round tray.
4. Place round tray onto autosampler, get a final sample count for end point and replace lid.
5. Make sure that round tray fits down flush onto the autosampler.
6. On computer screen, select “TOC-Control V” icon.
7. Then select “Sample Table Editor”
8. Enter user name: “analyst initials” select OK.
9. Under “File” select “calibration curve” “OK”.
10. Under system select Shimadzu TOC-BWS Enter/next
11. Select Edit Calibration points manually Enter/next
12. Under “Analysis” select “NPOC” then make up your file name (use today’s date) Enter/next.
13. Calibration Measurement Parameters are default: Just hit “next”
14. Select “ADD” and enter calibration points starting at (1) 100 mg/L (2) 50 mg/L (3) 25 mg/L (4) 10 mg/L (5) 5.0 mg/L (6) 2.5 mg/L (7) 1.0 mg/L (8) 0.0 mg/L. After 8 points it should show 0.00 mg/L first and 100 mg/L eighth if so “next”
15. Put a check mark in “Correlation Coefficient” check box “next”
16. “next”
17. “finish”
18. Go to file and select “new”, “sample run” “ok” “ok” enter file name: user date “save”
19. Now go to insert and select “calibration curve” then scroll till you find your file name/date should have .cal after date “select” the “open”
20. You should now see the sparging /acid addition page which shows a picture of the round sample tray. Under vial manually enter “1” beside 0.00 mg/L.
21. manually enter “2” beside 1.0 mg/L and “3” beside 2.5 mg/L and so on and so forth all the way to “8” this shows what order they are loaded on the tray. “Enter/OK”
22. Then a screen with your filename/date and all info should be in row 1 only with vial column showing. 1,2,3,4, etc.
23. Select the lightening bolt symbol then enter “use PC settings” this will start initializing wait till screen goes away then you will see the stop light symbol appear with green light showing, select that icon select “keep running” select “standby”
24. Sparging/acid addition page will re-appear just hit “OK”
25. Start ASI tray screen will appear hit “Start”
26. The instrument should start establishing the baseline and move auto tray into position – Lid must be on and samples loaded into correct position will take almost 3 hours to finish. Can view data as its coming off by selecting “view” “sample window”. After calibration is done review.
27. Select “File” then “New” then “sample run” “ok”
28. General information screen: No change select “ok”
29. Save as screen: Select today’s date for file name example 00month/00day/00year
30. Select “save”
31. Sample Table Screen: Select “insert” then select “auto generate” enter
32. **Page 1** sample group wizard sample source: select “calibration curve” then double click on box with 3 dots ...
33. Open latest curve from calibration curves file
34. Highlight latest curve and select “open”
35. Should send you back to page 1 with calibration curve info submitted. Select “next”
36. **Page 2** Sample Parameter: Enter final sample count for “number of samples” select “next”
37. **Page 3** Calibration Curves: No changes Select “Next”

38. **Page 4** Calibration Checks: No changes Select "Next"
39. **Page 5** Controls: No changes select "finish", Select "ok" on "Sparging/ Acid page.
40. Type sequence as they are loaded on tray: ICV, ICB, BSW, Sample #, client, etc.
41. Once everything is typed in double check that it matches the way samples and QC are loaded..
42. Click or select the lightening bolt symbol then select "use settings on PC". Wait for initializing. When screen goes away the traffic light symbol should appear next to the lightning bolt symbol. Click on the traffic light symbol.
43. Click or select "shut down Instructions". Then select "standby" Sparging/ Acid addition screen will appear so you can confirm your tray is loaded the wax things are highlighted in blue. Select "OK" if it looks the same.
44. Start ASI measurement: External acid addition should have a check mark click on "start" analysis should begin to start.
45. Click on view and chose "sample window" to watch curves come off and to see beginning values.

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TOC Data 2nd check

- ☐ Analyst authorization
- ☐ All general information complete
- ☐ Calibration meets criteria/ Date _____
- ☐ Base MDL
- ☐ Correct Units
- ☐ Corrections crossed out, initialed and dated, with reason (if necessary)
- ☐ Curve correlation & date good
- ☐ Dilution factors or Soil sample amounts correct
- ☐ TV's & sources indicated
- ☐ MRL performed and within control
- ☐ CCV/CCB done where required & in control
- ☐ BLK/BS per day & per 20 samples & in control (per 10 samples if 5310C)
- ☐ MS/MSD per 20 & in control
- ☐ MS prep included
- ☐ Blanks before samples all <DL
- ☐ 10% check of transcription from instrument
- ☐ Samples noted when <DL
- ☐ 2nd check of LIMS entry
- ☐ BS, MS/MSD prep. indicated &/or calculation shown
- ☐ Holding time met
- ☐ 9060 samples analyzed in quad
- ☐ One sample analyzed in quad per 20 for Lloyd Kahn
- ☐ Problems discussed with manager
- ☐ Necessary NCR's attached
- ☐ Sample _____ used for recalculation from raw data to final LIMS concentration
- ☐ Additional information needed for reports:

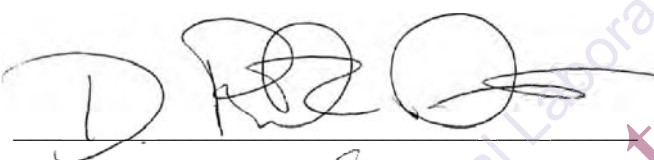
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STANDARD OPERATING PROCEDURE

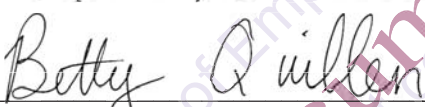
QUALITY SYSTEMS: QS08 REVISION #: 04 EFFECTIVE DATE: 20111031
Reviewed: 20130410 by Marcia K. McGinnity

**TECHNICAL / OPERATIONAL DEFINITIONS, MINIMUM ESSENTIAL
QUALITY CONTROL ELEMENTS, AND LABORATORY CALIBRATION
PROCEDURES (MAJOR & SUPPORT EQUIPMENT)**

APPROVALS:

Lab Director:  Date: 11/01/11

Data Quality Manager:  Date: 11/01/11

Section Supervisor:  Date: 11/01/11
Betty Quillen

Section Supervisor:  Date: 11/01/11
Jade Holliman

Reviewed 20130410 by Marcia K. McGinnity and found to need no revisions.

Change Summary – Revision 04, 10/31/2011.

- This SOP is an update to revision 03 dated 11/17/2010.
- LOQ evaluation criteria updated to reflect DoD QSM limits where available and default limits where not available with 20% allowance for marginal exceedences.
- References to DoD QSM 4.1 updated to reflect 4.2.

Change Summary – Revision 03, 11/17/2010.

- This SOP is an update to revision 02 dated 09/03/2010.
- Definitions for LOD and LOQ updated to include evaluation criteria.
- Table on page 12 updated to reflect purgeable organics with limits 50%-150%.

Change Summary – Revision 02, 09/03/2010.

- This SOP is an update to revision 01 dated 09/20/2009.
- DL/MDL/LOD/RL/LOQ definitions/relationships have been added/updated.
- Organic LOD criteria upper limit was changed to 200%.

1.0 SCOPE AND APPLICATION

- 1.1. Throughout the laboratory, various technical terminologies are used to communicate key quality control elements and the related data. This SOP is an attempt to provide a consistent format for all terminologies used and to define the laboratory's most commonly used technical terms.
- 1.2. As an analytical laboratory analyzing samples and providing environmental testing results, a critical element of data validation is calibration. This SOP provides guidance on the common protocols and elements to all methods performed by the laboratory.
- 1.3. The laboratory's technical SOPs will reference this Quality Systems SOP within section 13.0 of the SOP; however, any deviations or specific requirements outside the scope of this SOP will also be listed within section 13.0 of the technical SOP.
- 1.4. The NELAC, DoD QSM 4.2, various State & regulatory guidelines and workshops have been used as a basis for the technically sound practices, protocols, & policies that are outlined within this SOP as a basis for the laboratory personnel to perform their daily workflow.
- 1.5. Specific method SOPs will provide detailed requirements for application Quality Control samples along with the required frequency, criteria, and possible corrective actions to be followed for failures.
- 1.6. Many definitions within this SOP are listed using the NELAC standards and the DoD QSM 4.2 revision documents as references.

2.0 BATCH DEFINITIONS AND REQUIREMENTS

- 2.1 **Batch:** Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20-environmental samples of the same NELAC-defined matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24-hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An **analytical batch** can include prepared samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

2.1.1 Matrix Designation: The following matrix types are used to group samples in the laboratory:

- 2.1.1.1 Aqueous (Drinking Water, Ground Water, Waste Water, etc. and are reported on a wt/vol basis)
- 2.1.1.2 Solid (Soils and Sludges are reported on a dry wt/wt basis)
- 2.1.1.3 Wastes (Oils are reported on an wt/wt wet basis)
- 2.1.1.4 Additional matrix separation may apply for special Projects or for large sampling events. These may be applicable to saline or brine samples.

- 2.2 **Method Blank (Blk):** A Method Blank is an analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The Method Blank is used to assess for possible contamination during preparation and/or analysis steps.

2.2.1 The MB, for prepped samples, must be performed at a minimum of once per Batch.

- 2.2.2 For analytical procedures that do not involve a preparation prior to analysis, continuing calibration blanks, preceeding samples, will also serve as method blanks for associated samples.
- 2.2.3 Evaluate each MB based upon the criteria in the method SOP.
- 2.2.4 Corrective actions associated with contaminated Method Blanks are listed in SOP QS05.
- 2.3 Lab control Sample or Blank Spike (BS):** The BS is a controlled analyte-free matrix, which is spiked with known and verified concentrations of target analytes. Spiking concentrations can be referenced in the method SOP. The BS is used to evaluate the viability of analytes taken through the entire prep (when applicable) and analytical process.
- 2.3.1 The BS must be performed at a minimum of once per Batch (Prep or Analytical Batch). Per NELAC, exceptions to this rule would be for those analytes for which no spiking solutions are available such as Total Volatile Solids, pH, Color, Odor, Temperature, Dissolved Oxygen, and/or Turbidity; however, please follow the specific method SOP for specific QC requirements.
- 2.3.2 For analytical procedures that do not involve a preparation prior to analysis, the QCS and/or CCV will also serve as the BS for the twenty samples which follow.
- 2.3.3 For multi-component methods, use the following guidelines in selecting a spiking solution:
- 2.3.3.1 For methods with 1-10 parameters, spike all components.
- 2.3.3.2 For methods that include 11-20 targets, spike at least 10 or 80% of target, or whichever is greater.
- 2.3.3.3 For methods with more than 20-targets, spike at least 16-components.
- 2.3.3.4 Ensure that all targets are spiked at least once a quarter.
- 2.3.3.5 The DoD QSM requires that all target analytes be spiked.
- 2.3.4 Evaluate each BS based upon the criteria in the method SOP.
- 2.3.5 Corrective actions associated with failing BS are listed in SOP QS05.
- 2.4 Matrix Spike (MS) and Matrix Spike Duplicate (MSD):** This QC sample is used to assess the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. Spiking concentrations for the MS and MSD are outlined within each method SOP.
- 2.4.1 The MS & MSD shall be carried through the complete sample preparation and analytical procedure. The spiking occurs prior to sample preparation and analysis. Follow guidelines for method SOP's for the preparation & analysis of a batch MS & MSD.
- 2.4.2 An MS & MSD is performed per Batch of 20 samples. For certain methods, a matrix spike and a sample duplicate are acceptable in place of a MS & MSD. Please follow the method SOP for specific QC requirements.
- 2.4.3 The MS & MSD are calculated for percent recovery (accuracy). In addition to evaluating the accuracy for each matrix spike, the precision between the MS & MSD must also be calculated. Follow guidelines from SOP QS09 for calculations.
- 2.4.4 In cases where the accuracy or precision fails the specified control limit criteria, corrective action must be performed in accordance with SOP QS05.
- 2.4.5 Follow guidelines from SOP QS05 for applying data qualifiers to results.

2.5 Additional Prep or Analytical Batch Quality Control Definitions

- 2.5.1 Initial Calibration Blank (ICB):** Analysis of the Calibration Blank prior to the analysis of the daily calibration in order to assay the instruments response versus time at zero concentration. Follow the guidance of each method SOP for the analysis of this QC sample.
- 2.5.2 Initial Calibration Verification Standard (ICV):** A Standard solution used to verify the instruments response versus time at a concentration specified during calibration. This standard contains all analytes of interest for the applicable method and is analyzed prior to the analysis of any samples. Follow the guidance of each method SOP for the analysis of this QC sample.
- 2.5.3 Continued Calibration Blank (CCB):** Analysis of the Calibration Blank at a frequency stated in the method SOP. The purpose is to assay the instruments response versus time at zero concentration. Follow the guidance of each method SOP for the analysis of this QC sample.
- 2.5.4 Continued Calibration Verification Standard (CCV):** A Standard solution used to verify the instruments response versus time at a concentration and frequency specified during calibration. This standard contains all analytes of interest for the applicable method, and is analyzed prior to the analysis of any samples. Follow the guidance of each method SOP for the analysis of this QC sample.
- 2.5.5 Initial Performance Check (IPC):** A solution of selected method analytes used to evaluate the performance of the instrumental system with respect to a defined set of method criteria. Follow the guidance of each method SOP for the analysis of this QC sample.
- 2.5.6 Tune:** Tune evaluation is a way of verifying the performance of the instrument. This is done by analyzing a sample of the tune evaluation mix and then evaluating a resulting mass spectrum for mass calibration, mass resolution, and/or ion ratios based on criteria specified in the environmental analysis protocol as stated in the method SOP.
- 2.5.7 Quality Control Standard (QCS):** The solution of method analytes of known concentrations that is used to fortify an aliquot of reagent water or other blank matrix. The QCS is a controlled analyte-free matrix, which is spiked with known and verified concentrations of target analytes. Spiking concentrations can be referenced in the method SOP. The standard used in the preparation of the QCS is obtained from a source external to the laboratory and different from the source of calibration standards. The QCS is used to check laboratory performance with externally prepared test materials. Follow the guidance of each method SOP for the analysis of this QC sample.
- 2.5.8 Sample Duplicate (DUP):** Reanalysis of a sample, usually immediately following the parent sample analysis. The results from the two analyses are compared to ensure that repeated analyses of this matrix will yield comparable results.

3.0 ADDITIONAL TECHNICAL DEFINITIONS

- 3.1 Calibration Range:** Calibration Range: The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.
- 3.2 Confirmation:** Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:
- ☐ Second column confirmation;
 - ☐ Alternate wavelength;
 - ☐ Derivatization;
 - ☐ Mass spectral interpretation;
 - ☐ Alternative detectors; or
 - ☐ Additional cleanup procedures.
- 3.3 Corrective Action:** The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.
- 3.4 Demonstration of Capability:** A procedure to establish the ability of the analyst to generate acceptable accuracy.
- 3.5 Detection Limit (DL):** The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. (NELAC) This definition is further clarified in the DoD QSM 4.2 revisions as the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.
- 3.6 Holding Times (Maximum Allowable Holding Times):** The maximum times that samples may be held prior to analysis and still be considered valid or not compromised.
- 3.7 Internal Standard:** A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.
- 3.8 Interference, chemical:** Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte.
- 3.9 Interference, spectral:** Occurs when particulate matter from the atomization scatters the incident radiation from the source or when the absorption or emission of an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible.
- 3.10 Limit of Detection (LOD):** An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent. This definition is further clarified in the DoD QSM 4.2 revisions as the smallest amount or concentration of a substance that must be present in a

sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%. This value is verified quarterly for all analytes and surrogates with a spiked standard taken through all preparation/analysis steps confirming detection at the specified concentration.

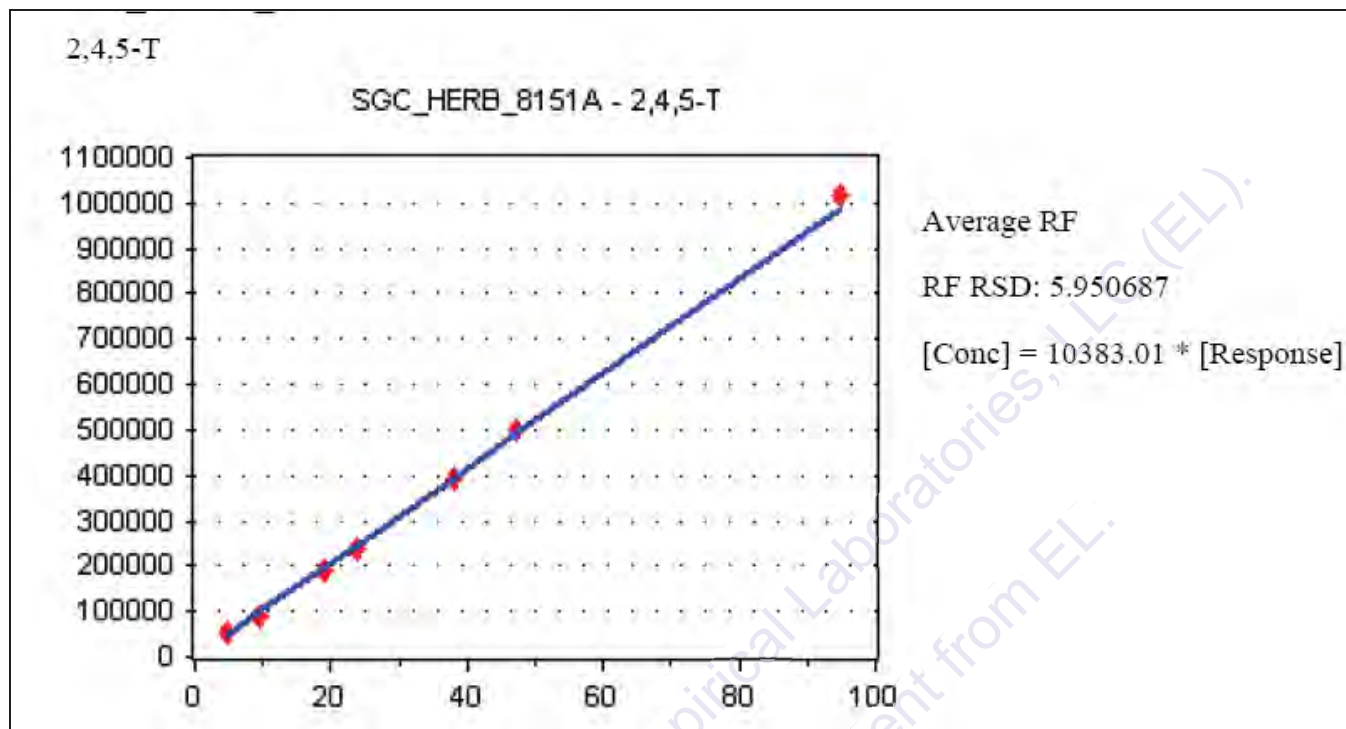
- 3.11 Limit of Quantitation (LOQ):** The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. This term is further clarified within the DoD QSM 4.2 as the lowest concentration that produces a quantitative result within specified limits of precision and bias. The LOQ shall be set at or above the concentration of the lowest initial calibration standard. This value is verified quarterly for all analytes and surrogates with a spiked standard taken through all preparation/analysis steps confirming acceptable recovery at one-half to four times the specified concentration. DoD QSM limits are applied, where available. Default limits (where QSM limits are not available) are 50%-150% for inorganics/purgeable organics and 10%-200% for extractable organics. DoD QSM marginal exceedence (ME) limits will be accepted for up to 20% of the DoD analytes with metals ME 75%-125%.
- 3.12 Method Detection Limit (MDL):** The constituent concentration that, when processed through the complete method, produces a signal with a 99% probability that it is different from the method blanks. This is generally interpreted to mean that the response, instrument signal or otherwise, for the MDL concentration is reliably greater than that for the method blank. It is important to note that the MDL is highly matrix dependent. Due to the numerous matrices possible, the MDL is typically determined in reagent water and analyte free soil. When this is the case, the MDL should be referred to as the “reagent water method detection limit” (RWMDL), and should not be applied to any other sample matrix without notation. The method detection limit, reagent water or otherwise, should not be confused with instrument detection limit (IDL) or lower limit of detection (LLD) which do not include the sample preparation portion of the method. Studies for MDL’s will be performed at initial setup for all method, analyte, & matrix combinations performed in each department and validated annually, at a minimum.
- 3.13 Nonconformance:** An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.
- 3.14 Raw Data:** Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.
- 3.15 Reporting Limit (RL):** A client-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix (DoD QSM 4.2). For a target parameter, the laboratory will analyze at least one point of the calibration curve at the Reporting Limit. Any exceptions to this rule are specified within the technical method SOP.

- 3.16 Retention Time:** The time between sample injection and the appearance of a solute peak at the detection.
- 3.17 Signal to Noise Ratio:** The signal carries information about the analyte, while noise is made up of extraneous information that is unwanted because it degrades the accuracy and precision of an analysis and also places a lower limit on the amount of analyte that can be detected. In most measurements, the average strength of the noise is constant and independent of the magnitude of the signal. Thus, the effect of noise on the relative error of a measurement becomes greater and greater as the quantity being measured (producing the signal) decreases in magnitude.
- 3.18 Spike:** A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes (DoD QSM 4.2).
- 3.19 Surrogate:** A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.

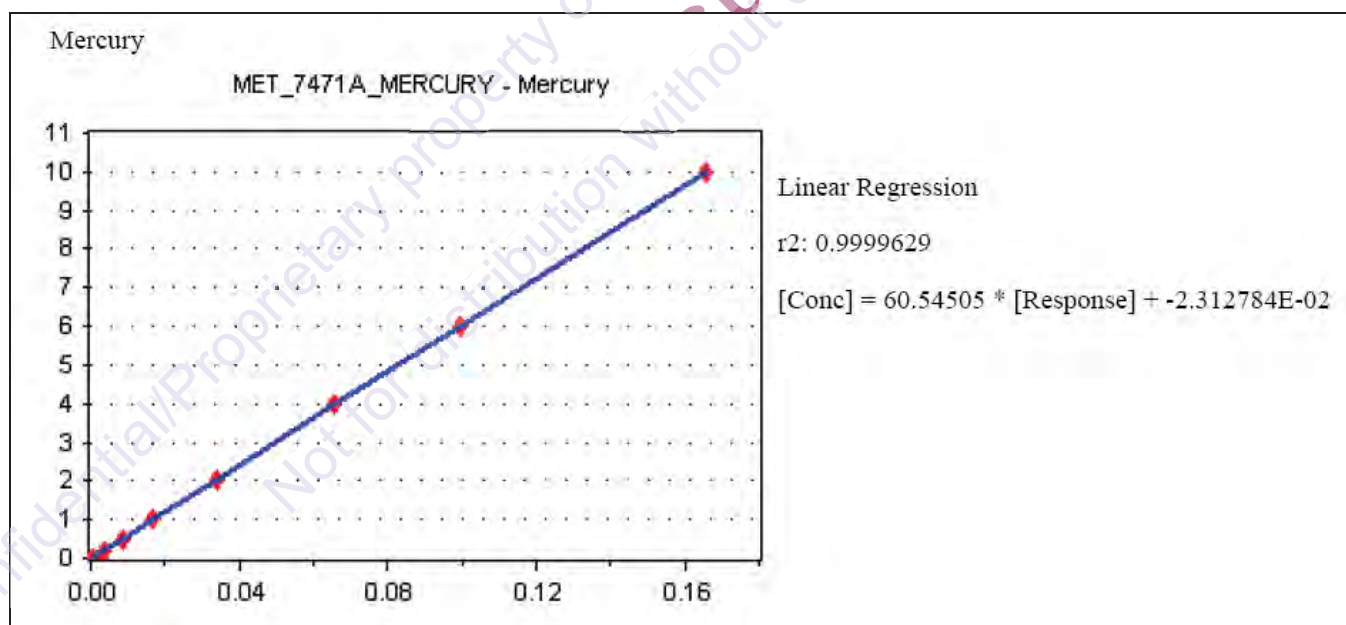
4.0 CALIBRATION

- 4.1** Calibration is the determination of the ratio of analytical response to analyte concentration. Calibration Curve: The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. Processes used could include titrimetric & gravimetric methods amongst others. Instruments used for analysis are calibrated prior to the analyses of samples. It is the laboratory's protocol that only first and second order fits be used to achieve calibration requirements. Higher fits may be utilized when necessary; however, when this occurs, the calibration curve data must be reviewed and signed by the department supervisor. The correlation coefficient may not be less than 0.995. The laboratory's technical method SOP's list the calibration criteria which is acceptable for the analyses of samples. An alternative to a linear calibration fit is using the option of Average Response Factor. Please follow the technical method SOP for protocols on using the Average Response Factor.
- 4.2** It is unacceptable to drop calibration points in the middle of the calibration range; however, levels at the high or low end may be dropped with specific technical explanations such as, instrument saturation, poor response, etc. Should specific mid level calibration levels need to be dropped, they must be dropped for all the parameters, with specific technical clarifications (poor injection, etc.), and with supervisory approval.
- 4.3** When employing a higher order fit, sufficient data points must be used to clearly define the calibration range. Analysts and supervisors must ensure that all portions of the curve have sufficient slope to assure adequate sensitivity and accuracy for the analysis.
- 4.4** Calibration standards are a set of solutions prepared at various concentrations. These standards are then analyzed to define the calibration range and calibrate the analytical process. Figure 1 is an example of an Herbicide SW8151A analyte's calibration & Figure 2 is an example of Mercury calibration.

Example Calibration (Figure 1)



Example Calibration (Figure 2)



- 4.5 When a **QCS** standard is employed, that standard is prepared from a source different than the curve source and prepared at approximately the low to mid-level of the curve. This standard is calculated against the calibration curve to verify the accuracy of the calibration curve source.
- 4.6 Continuing calibration verification standards (**CCV**) are run at method required intervals to validate the calibration. When the CCV standards pass required criteria, the system is considered to be within calibration and the analysis of unknown samples can continue. However, when the CCV standards fail criteria, corrective actions must be implemented

which may include maintenance of the system, re-analysis of the CCV, and/or re-calibration of the analytical system.

5.0 Batch Evaluation

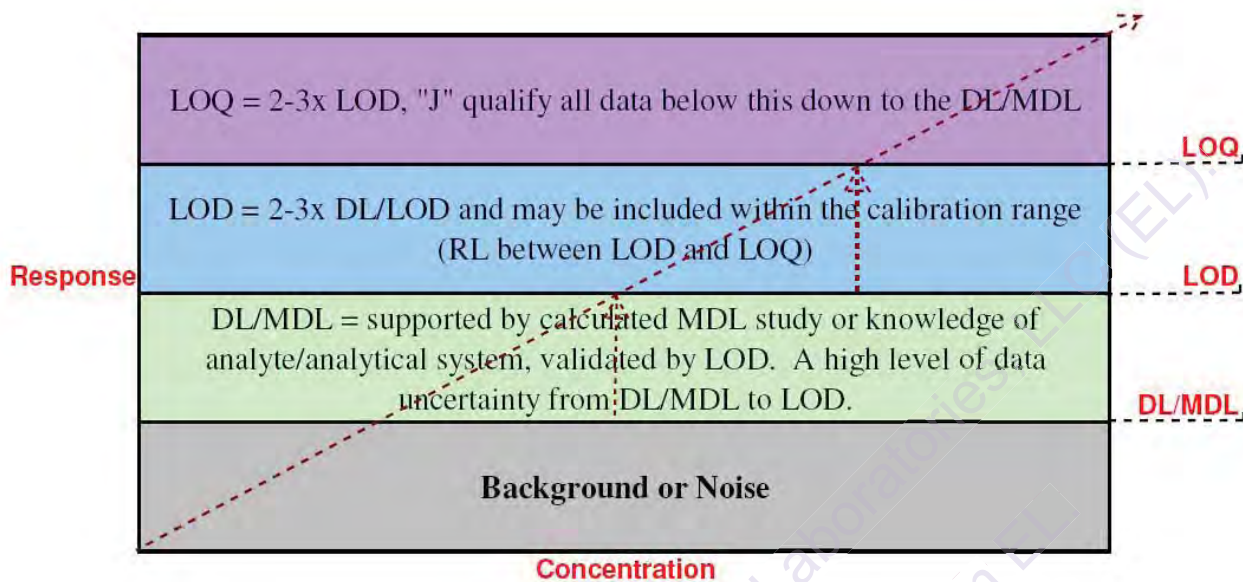
5.1 Several elements must be evaluated when analyzing all components of a Batch.

5.1.1 **Completeness:** An evaluation of the chemical measurements (results) are accomplished by evaluating spike & matrix spike accuracy and precision (SOP 10-11), when applicable surrogate and internal standard recovery (method specific SOPs), sample and sample duplicate result replication (method specific or client specific guidelines), and the evaluation of the Method Blank (SOP 10-04 and method specific SOPs). Please reference SOP 10-04 when any required element of a Batch fails.

5.1.2 **Comparability:** defines the extent to which a chemical parameter measurement is consistent with, and may be compared to values from other sampling events. When there is an on-going sampling event, results can be evaluated against historical data. This evaluation may encompass the analytical and the project management group.

6.0 RELATIONSHIP BETWEEN LAB MDL/DL, RL, LOD, & LOQ

- 6.1 The laboratory's LIMS system lists each of the values reported on the Final Report. Due to the various programs to which final reports are provided, and because of various terminologies and program definitions, the laboratory is providing the relationship of various reporting terms.
- 6.2 The lab reported DL (MDL) is generally based upon a full MDL study but may be based upon knowledge of the analyte/analytical system with validation of detection at the LOD.
- 6.3 The lab reported LOD is usually 2-3 times the DL (MDL).
- 6.4 The RL can be set to a value between the LOD and the LOQ but shall be set at or above the concentration of the lowest initial calibration standard or verified with a standard at the concentration of the RL.
- 6.5 The lab reported LOQ is usually 2-3 times the LOD but may be equal to the LOD.



Description / Requirement	LIMS – MDL Column	LIMS – LOD Column	LIMS – MRL Column
Definition	The lab reported DL (MDL) is generally based upon a full MDL study but may be based upon knowledge of the analyte/analytical system with validation of detection at the LOD.	Per SOP definition, may be the same as the RL	Per LOQ definition
Frequency	At method/analyte setup then validated annually at LOD except for DoD where the LOQ & LOD checks are required quarterly	Quarterly	Quarterly
Criteria to Pass	<ul style="list-style-type: none"> ➤ Calculated MDL value must be greater than 10% of the Spike Level ➤ Calculated MDL value must be less than the Spiked concentration ➤ Preparation/Analysis at LOD must produce detectable results. 	<ul style="list-style-type: none"> ➤ Parameter must be detected ➤ Check for Ion Abundance on MS methods ➤ 2nd column / detector confirmation is required ➤ Pattern recognition is needed ➤ Response must be 3-times the noise level 	Bias Requirement <ul style="list-style-type: none"> ➤ DoD QSM limits, where available. Default limits (where QSM limits not available): <ul style="list-style-type: none"> ➤ Inorganics 50%-150% ➤ Purgeables 50%-150% ➤ Extractables 10%-200% ➤ QSM Marginal Exceedence (ME) limits accepted for up to 20% of QSM analytes with metals ME 75%-125%. ➤ The LOQ value must be greater than or equal to the LOD value
Number of Standards	7-10 replicates	1-standard	1-standard
Prep Requirement	Prepped per matrix	Prepped per matrix	Prepped per matrix
Analysis Requirement	Analysis per instrument or validation with LOD on similar instrument configuration.	Analysis per instrument	Analysis per instrument

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

QUALITY SYSTEMS: QS10

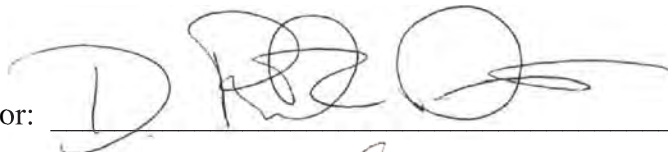
REVISION #: 20

EFFECTIVE DATE: 20130903

LABORATORY SAMPLE RECEIVING, LOG IN AND STORAGE

APPROVALS:

Lab Director:



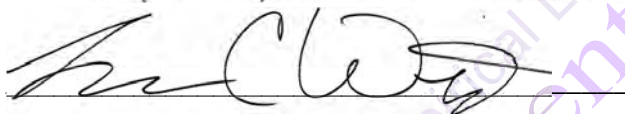
Date: 09/03/2013

Data Quality Manager:



Date: 09/03/2013

Group Leader:



Date: 09/03/2013

Changes Summary

Revision 20, 09/03/2013:

- Sections 2.5.3.4 and 7.3 referencing Aquatic Toxicology samples removed.
- Excluded oil and grease from pH checks in section 3.3.1.1 and removed reference to oil and grease in section 3.3.2.3
- Added section 3.3.6 requiring that any sample adjustment be noted within the LIMS.
- Updated section 4.1.1 to reflect VOC Water Refrigerator, added listing for segregated VOC Soil area and updated section 8.2.3 to indicate segregated VOC soil area.
- Placed all VOC storage prior to water and soil walk-in except for VOC Dry Storage Rack. Added Storage Blank reference to all VOC cold storage areas.
- Added Truck Maintenance Log to list of log books maintained by SR.
- Updated Attachment references throughout document and updated references to Sample Change Form to indicate e-mail notification.
- Removed list of holding times and preservations - added reference to table in QS11 in section 2.6.2.
- Removed Container Codes Attachment for the LIMS and "Green Sheet" for LIMS corrections (Old System).
- Updated Soil Movement Map and Custody Log. Added maps specific to fire ants and nematodes.

Revision 19, 09/17/2012

- Removed section 3.3.2.7
- Updated section 5.2 and 9.1.2 to reflect second checking of primary login information with update to SRC form.
- Updated personnel references to reflect Sample Receiving Personnel, Group Leader, Section Supervisor, Project Manager and Lab Director.
- Removed information for thermometer calibration requirement in section 2.4 and container preservation/traceability in section 3.3.3, as they are covered in other SOPs.
- Removed redundant statements from section 2.1, 3.3 and 3.4.
- Reference to sample number reset at the beginning of the year removed from section 3.2.
- Removed reference to pH paper method 9041A in section 3.3.3.2.
- Added section 3.3.4.5.3 requirement to note date/time of preservation in comment section.
- Removed reference to Na2S2O3 preservation in section 3.3.6.
- Updated refrigerator tolerance range to 0°C-6°C in section 4.5.
- Removed reference to back up compressor in section 4.6.
- Updated sample retention to 45 days from sample receipt as is consistent with all proposal notes.
- Section 5.1.2 updated to add **"Note – correct/accurate selection is absolutely critical to the successful analysis, reporting and invoicing of all samples."**
- Section 5.1.19 updated to make sample receiving personnel contact PM when appropriate test code is not available in the selected project.
- Reference to temperature monitoring of the walk-in coolers has been updated in section 4 to include TempAlert system.
- Section 6.4 updated to remove items associated to previous LIMS system.
- Log book IDs removed from section 7.4.
- Sample Receiving Custody and Disposal Form removed from SOP as Custody is maintained in the LIMS system and disposal is handled separately.
- Attachment identifications updated and Short Holding Time Parameters attachment replaces with List of Holding Times/Preservations.

Revision 18, 09/16/11

- The cooler receipt form was revised.
- Added requirement that samples must be refrigerated within 2 hours of opening cooler.
- Added cooler receipt form completion requirement upon opening cooler.
- Updated procedure for pH measurement.
- Changed default time to 00:01 in the case a time is not listed on COC.
- Removed reference to ATSD in SOP 187.

Revision 17, 05/16/11

- The list of employees has been removed from section 8.

Revision 16, 11/17/10

- Added requirement “Notes to sample analytical comments indicating date/time/initials preserved.” for metals samples preserved within house.

Revision 15, 10/13/10

- Updated CRF on page 23, also added at the beginning of page 6 statement about recording the final temperature value.

Revision 14, 09/07/10

- The SOP combines SOPs 404, 406, 410, 415 and 432 into one SOP with updated naming.

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Table of Contents

1. Sample Acceptance Criteria
2. Sample Receiving
3. Sample Log In
4. Sample Storage
5. Laboratory Information Management System (LIMS)
6. Daily Follow Up for Sample Receiving/Log In
7. Miscellaneous
8. Sample Storage, Secure Areas, and Sample Custody
9. Sample Receiving Personnel Duties and Responsibilities
10. Procedure for Treatment of Soil Samples from Quarantined Areas
11. Subcontracting Laboratory Samples
12. Attachments

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1.0 Sample Acceptance Criteria

This SOP lists, in as much detail as possible, our daily procedures for sample receiving, log in and storage of laboratory samples. Keep in mind that there may be project specific requirements that are more strict or different than our routine procedures. In these instances, the project specific requirements must be met and followed. Although a few project specific requirements are detailed in this SOP, i.e. DoD requirements, not every situation can be addressed. If there is ever any uncertainty on what procedures must be followed, please see the Project Manager or your Section Supervisor immediately. If ever in doubt, always go with the more stringent requirements.

- 1.1 A sample may be rejected for compliance purposes if it does not meet the following criteria. Analyses may only proceed after notification and approval to proceed from the client or from the laboratory director.
 - 1.1.1 Sample must be properly preserved and in the proper container for the requested analysis.
 - 1.1.2 Sample integrity must be maintained. The container shall be intact without cracks, leaks, or broken seals.
 - 1.1.3 Adequate sample volume must be received for the requested analysis, including volume for any requested QA/QC (MS/MSD).
 - 1.1.4 The sample ID on the bottle label must match the sample ID listed on the chain of custody.
 - 1.1.5 The sample container label and the chain of custody must be completed with indelible ink. The sample label must be intact and list all necessary information; to include: sample date, sample time, and sample ID/location. The chain of custody shall also indicate sample date and time, requested analyses, and all necessary client information.
 - 1.1.6 Sample temperature must be less than 6°C or received on ice.
 - 1.1.7 Sample must be within holding time for the requested analysis.
 - 1.1.8 Samples should be refrigerated within 2 hours of being unloaded.These issues are discussed in more detail below under the “Sample Receiving” section of this document.

2.0 Sample Receiving

- 2.1 Samples are received at the Empirical Laboratories on 621 Mainstream Drive, Suite 270 Nashville, TN 37228.
 - 2.1.1 The majority of samples are shipped in coolers by couriers such as Federal Express and UPS. All couriers are generally received in the Empirical Laboratories Sample Receiving (SR) area loading dock in back of the laboratory. The laboratory is located close to the Federal Express (FedEx) distribution station, therefore we do pick up our coolers at the FedEx location and transport them back directly to the laboratory.
 - 2.1.2 Some coolers and/or samples may be received directly by Empirical Laboratories Sample Receiving personnel. If samples are hand delivered by the client make sure that necessary paperwork is included and that you sign and date the chain of custody, as well as record the final value temperature of the samples on the chain of custody as well. If the *Empirical Laboratories Chain of Custody [Attachment I]* is used the white and yellow copy of the chain of custody is retained and the pink copy must be given to the client.
- 2.2 Sample receiving personnel must wear the following personal protection equipment: gloves, safety glasses and a laboratory coat.

- 2.3 Visually inspect all coolers for tampering, custody seals, (intact if applicable) leakage, etc. If a cooler has been damaged beyond repair, unpack the samples and discard the cooler as to not reuse it. If you suspect a cooler may be damaged or is extremely dirty this cooler must not be reused. If coolers were sent by Federal Express, examine the Federal Express air bills for the number of packages in the shipment and make sure that all the packages (coolers, boxes etc.) in a group have been received. If there are any problems the Project Manager must be contacted immediately. If anything looks unusual, take the time to check it out and document the situation and findings.
- 2.4 Each cooler must be opened and a cooler receipt form must be completed and placed in cooler. Open each cooler in order to quickly inspect the contents and to locate the chain of custody. Your signature and the date and time the samples were received must be placed onto the chain of custody. The time received must reflect the actual time the samples were received even though they may be logged into the system at a later time. Samples received late in the day during the week may be processed the next morning. All cooler(s) must be opened, examined (for leakage, breakage etc.), the temperature measured and the chain of custody signed and dated to reflect the actual date and time which they were received. The samples must be delivered to the appropriate analytical department or put in cold storage as soon as possible after unloading.
- 2.4.1 Attach any shipping receipts, work orders, documentation, etc. to the chain of custody.
- 2.4.2 If a chain of custody or other paperwork is not sent, the client must be contacted and the samples temporarily placed on hold in cold storage. The required information may be found on the sample containers or it may be necessary to call the client to get the missing information (i.e. sample ID, collection date and time, etc.).
- 2.4.3 Project specific paperwork may be required. For all projects, a *Cooler Receipt Form [Attachment II]* must be completed for each cooler received. Sample receiving personnel must begin completing this form as soon as a cooler is received and complete this form as samples go through the log in process.
- 2.5 The temperature of each cooler or set of samples must be measured as quickly as possible using a thermometer with 0.1°C increments.
- 2.5.1 To measure the temperature, point the IR gun at the cooler temperature blank (if supplied) or a direct sample and wait a few seconds for the temperature to stabilize. The IR gun should be held 6 inches away (from the temperature blank or sample) for an accurate reading. Read the temperature to the nearest 0.1 °C. The final value temperature value must also be recorded on the chain of custody. (This value will also be recorded into the LIMS at a later point.). This is calculated by measuring the initial value temperature and adding the correction value temperature (IR temperature gun calibration factor) together, to obtain the final temperature value. All regulatory compliance samples received from North Carolina that do not meet the temperature requirement will be segregated and the client will be notified of the non-compliance. The samples will not be analyzed until we receive client notification to proceed with analyses.
- 2.5.2 If the temperature exceeds 6°C for any sample, the Project Manager must contact the client immediately. There may be tighter temperature control limits for specific project requirements. The customer must make the decision to either continue with the analyses or resample. Make sure the client is aware that temperature exceedence will be narrated if the samples are analyzed. Not all samples for the project will be narrated, just those samples received above 6°C.

Note: Many times we are not able to get in touch with the client quickly and the best judgment on how to handle the samples must be made after discussion with the Project Manager and/or Laboratory Director. The samples may still need to go through the log in process although it may be eventually determined that the samples will not be analyzed or the samples may temporarily be placed on hold and not logged in, with the exception of short hold and rush samples. **Above all, do not allow the samples to set out at room temperature for an extended period of time while waiting for a decision.**

2.5.3 The only exceptions to the 6°C rule are:

2.5.3.1 Water samples for all Metals, (except Chrome 6+ and mercury) that have been preserved with HNO₃ to a pH of ≤ 2 . *Keep in mind that non-aqueous sample for Metals must be cooled.*

2.5.3.2 Samples for Fluoride, Chloride and Bromide.

2.5.3.3 Waste/Product samples for all parameters.

2.5.3.4 Samples collected locally by Empirical Laboratories personnel or hand delivered by local customers. In some instances these samples may not have had time to cool down; however, these samples should have been placed on ice in an attempt to cool them to the proper temperature. This exception is only applicable if the samples were collected the same day as the laboratory receives them. It should be noted if samples are "Received on Ice" (ROI).

2.6 If several coolers are received at once, they must be inspected to determine the order in which the samples should be unpacked and logged in. The following priorities should be given:

2.6.1 Hexavalent chromium water samples which have a 24 hour holding time.

2.6.2 Any analyses which have a 24-72 hour holding time (See QS11 for table).

2.6.3 Any sample which has almost exceeded its' holding time. (Especially watch for this with waters organic extractions, Solids and Sulfides, all of which have only 7 days). A list of parameters and holding times is posted in sample receiving from QS11.

2.6.3.1 If a sample is received already out of holding time, this must be documented and the Project Manager must be contacted. The sample can be analyzed at the client's request, but it will be qualified on the final report as being received out of holding time. The Project Manager will inform you of the client's decision.

2.6.3.2 If a sample is received with limited holding time remaining for any parameter, it is necessary to contact the Project Manager so that he/she can contact the client. If the sample has to be analyzed on a rush basis to meet the holding time, a rush charge may apply. Also it may not be possible to analyze the sample within the holding time due to sample load, etc.

2.6.4 Samples requiring rush turnaround.

2.6.4.1 If sample(s) require 24 or 48-hour turnaround they will take first priority. Other rush requests also have high priority.

2.6.4.2 The Project Manager and/or Section Supervisor must be contacted for approval concerning any unscheduled rush requests.

2.7 Unpack all samples from the cooler. If there are any known or suspected hazards this must be done under a hood. It may be necessary to rinse off the outside of the containers in the sink and/or wipe them off with a paper towel.

- 2.7.1 Visually inspect them for tampering and custody seals (if applicable). Sort and inventory the samples against the chain of custody by arranging them in the same order as they are listed on the chain of custody. Normally samples are assigned log numbers in the same order as they are listed on the chain of custody but for certain projects or situations it is acceptable to arrange them in a manner which will make them easiest to log in.
- 2.7.2 Check for leakage and sample container breakage as this could compromise the sample integrity. If any spillage occurred in the cooler make sure this is noted. Also list all the other samples in the cooler as cross contamination could occur. The Project Manager and/or the customer may need to be notified in these situations. It may be necessary to resample.
- 2.8 Check the chain of custody information against the information recorded on the containers. If these do not agree, this must be documented and the Project Manager must be notified.
 - 2.8.1 If major changes are made on the chain of custody received from an engineering job, then the client should submit written confirmation of these changes or make the corrections and initial them directly on the chain of custody.
 - 2.8.2 Any error found on the chain of custody must be marked through with one line, initialed, dated and the correction written in.
- 2.9 Note any unusual requests, methodology, hazards (known or suspected) to the Project Manager and/or Section Supervisor or analysts before the samples are actually logged in. Make notes of any problems (improper containers, preservatives, temperature, or descriptions, etc.).

3.0 Sample Log In

- 3.1 After samples have been unpacked, sorted and reviewed, they are then ready to be assigned log numbers and continue through the log in process. Make sure that the parameters for the samples are clearly marked on the chain of custody. Contact the Project Manager if there are any questions, problems, etc.
- 3.2 Assign a work-order and sample number to each individual sample and record it on each sample container and the chain of custody
 - 3.2.1 All containers with the same description must have the same sample number even if they have different preservatives and require different tests. However, each different fraction (bottle type and/or preservative) should be designated with a letter (A, B, C, etc.).
 - 3.2.2 Grab and composite samples from the same sample location must be considered as separate samples. It may be necessary to use "grab" or "composite" as part of the sample description to distinguish between the samples. Only assign different log numbers to them if the parameters are clearly marked as grab and as composite. Do not assume that VOC must be analyzed from grab samples so therefore the client must have taken a grab sample.
- 3.3 Check the following items and record this information on the cooler receipt form to further ensure sample integrity. If any of the following requirements are not met it may be necessary to contact the client. We can perform the analyses in most cases and will do so with the client's approval, however the results may be qualified in some manner on the final report.

Preserving sample integrity throughout the log in procedure must be one of our section's top priorities. This includes not only ensuring that the proper chemical preservatives have been added but also that the samples are received and maintained at the proper temperature.

Samples should not set out at room temperature if there is a delay. The samples must temporarily be placed in cold storage until you are able to complete the log in procedure. **[Make sure the VOC containers are not temporarily stored in a non designated VOC only storage area.]**

3.3.1 Check to determine if the proper chemical preservatives were added to adjust the sample to the correct pH. Any regulatory compliance sample received from North Carolina that does not meet the preservation requirement will be segregated and the client will be notified of non-compliance. The samples will not be analyzed until notification to proceed with analyses is received from the client. A list of parameters and the required chemical preservatives is posted in the log-in room. The verification of this preservation will be recorded on the Cooler Receipt Form for all projects.

3.3.1.1 The pH of each container (except VOA vials or Oil and Grease samples) which requires pH preservation must be checked. Do not open and check the pH of VOA vials or Oil and Grease containers in sample receiving/log-in. This information is then documented on the project cooler receipt form.

3.3.1.2 The pH of preserved samples is checked and confirmed using pH narrow range indicator paper.

3.3.1.3 When taking the pH reading, DO NOT PUT THE pH PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Use capillary tube to measure pH. For some samples (wastes) the indicator paper may not be accurate due to interferences. The observation of the appropriate color change is a strong indication that no interferences have occurred. If it appears as if there is interference, the pH must be measured using the pH meter. [See SOP187 pH, Electrometric.]

3.3.2 The following guidelines must be followed to check pH preservation:

3.3.2.1 Water samples for Cyanide analyses must be preserved to a pH of >12.0 with NaOH upon collection. If the pH of these samples is <12.0 upon receipt, the client must be notified immediately. Upon client approval, the sample should then be adjusted to >12.0.

3.3.2.2 Water samples for Metals analyses must be preserved to a pH of <2.0 with HNO₃ upon collection. If the pH of these samples is >2.0 upon receipt, the client must be notified immediately. Upon client approval, the sample should then be adjusted to <2.0.

3.3.2.3 Samples requiring analyses which are preserved with H₂SO₄ (i.e., Nitrogen compounds, Total Phenolics, Total Phosphorus, etc.) should be preserved to have a pH of <2.0. If the pH of these samples is >2.0 upon receipt, the client must be notified immediately. Upon client approval, the sample should then be adjusted to <2.0.

3.3.2.4 Samples for sulfide analysis must have a pH >9.

3.3.2.5 If a sample is not properly preserved, log-in personnel must do one of the following:

3.3.2.5.1 To meet project specific requirements, the client must be notified before preserving or adding additional preservative to the sample unless otherwise instructed. If the client instructs us to add chemical preservatives to a sample, complete traceability of the preservatives used is required.

- 3.3.2.5.2 For other projects it may be acceptable to preserve the sample accordingly before the sample is placed in storage. Complete traceability of the preservatives used is required.
- 3.3.2.5.3 All metals samples preserved upon receipt must be held 24 hours before proceeding with analysis (indicate date/time of preservation in comment section). The client must be notified to see if the lab is to proceed with analysis. Notes to sample analytical comments indicating date/time/initials preserved.
- 3.3.2.6 In some instances it may not be possible to adjust the sample to the proper pH due to matrix problems which cause excessive foaming or require an unusually large amount of acid. Do not continue to add acid if a few mL's of acid does not lower the pH. Notify the Project Manager, Metals Section Supervisor and/or analyst. They will make the decision if the sample will be diluted, not analyzed, etc. Make sure you note on the cooler receipt form and in the LIMS notes that the sample is not at the proper pH as well as any useful information (i.e., foaming, strong odor, etc.).
- 3.3.3 Check to make sure samples are in proper containers and that there is adequate volume for all the parameters requested and no leakage.
- 3.3.4 If VOA vials are present, each vial must be inverted and checked for head space. "Pea-sized" bubbles (i.e. bubbles not exceeding 1/4 inch or 6 mm in diameter) are acceptable and should be noted. Large bubbles or head space is not acceptable and this information must be documented on the cooler receipt form. If this occurs, the client must be contacted. The samples can be analyzed with their approval, however the report will be qualified and the data may be questionable.
- 3.3.5 All chlorinated effluent samples received for Cyanide must be checked for residual chlorine. The one liter sample container should initially contain 1 to 2g/L of Ascorbic Acid. Potassium Iodide starch indicator paper will be used for detecting the presence of residual chlorine. DO NOT PUT THE TEST PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Use capillary tube to test and dispose of this volume after the sample is checked. If the test paper turns blue, the sample must be treated for residual chlorine. Add ascorbic acid approximately 0.6g at a time and recheck the sample until there is no residual chlorine present. If the sample required this treatment this information must be included on the cooler receipt form. This must be done by log-in personnel before leaving the receiving area. It may be necessary to notify the Inorganic Section Supervisor and/or analyst.
- 3.3.6 Any sample adjustments MUST be noted within the LIMS.

4.0 Sample Storage

- 4.1 After samples have been correctly logged in they are then transferred to one of the following cold storage areas and arranged in numerical order by the assigned log in/LIMS sample number. ***Note that solid and aqueous VOC samples must be segregated from all other samples.***
 - 4.1.1 VOC Water Refrigerator: All aqueous VOC's must be stored in this refrigerator. Storage blanks consisting of organic free water from the laboratory may be required for specific projects. These will be analyzed for VOCs only. ***Storage blanks are required for all DOD projects.***

- 4.1.2 VOC Soil Freezer: All soil samples requiring VOC analysis with short hold prep times (Encores, Organic Free Water Terracores, etc.) must be stored in this freezer. ***Storage blanks are required for all DOD projects.***
- 4.1.3 VOC Soil Segregated Area: All bulk or chemically preserved soil samples requiring VOC analysis must be stored in this area. ***Storage blanks are required for all DOD projects.***
- 4.1.4 Walk in Refrigerator: All aqueous samples for all other analyses must be stored in this refrigerator.
- 4.1.5 Soil Walk-In Refrigerator: All quarantined and non-quarantined soil samples for all analyses must be stored in this refrigerator.
- 4.1.6 VOC Dry Storage Rack: All water VOCs that have exceeded double holding time can be stored on this rack. These samples are stored here segregated alone to ensure no cross contamination occurs between VOC samples and other non-VOC aqueous samples.
- 4.2 Quarantined soils are those quarantined by the US Department of Agriculture. A separate disposal log must be maintained for these soils including the location, date and quantity of the soil received and processed. Soil residues from quarantined samples must be treated according to regulations after testing. Quarantined soils are defined as:
 - 4.2.1 Soil taken from much of the southeastern US and parts of New York and Maryland at a depth of three feet or less. *Soils from three feet or more are not regulated provided they are stored separately.* A map of the regulated areas in the United States entitled ***Soil Movement Regulations [Attachment III]*** is posted in the log-in room.
 - 4.2.2 All soils taken from foreign sources, US Territories and Hawaii.
NOTE: All soils are treated as quarantined soils and are disposed of in accordance with USDA regulations. Above for information purposes only.
- 4.3 All samples must be stored in one of the four refrigerators detailed above with the following exceptions:
 - 4.3.1 Matrices that may be adversely affected by the cold temperature. (E.g. surfactant samples, multi-phase samples).
 - 4.3.2 Highly contaminated waste or product type samples that could jeopardize the integrity of other samples in the walk in cooler. Often these can be stored at room temperature. If these require refrigeration see the Project Manager for other options.
- 4.4 The temperature of each sample refrigerator is monitored and recorded each day. A Mercury thermometer or digital min/max thermometer with 1° increments must be used. Each thermometer must be calibrated against a NIST certified thermometer once a year (**digital thermometers quarterly**). The thermometers must be tagged with a unique identification, the date calibrated and the correction factor.
- 4.5 The tolerance range for all refrigerators is 0°C to 6°C. If the temperature exceeds this range, corrective action measures must be implemented immediately. The Section Supervisors, Data Quality Manager and Laboratory Director will be notified in order to assess the situation. It may be necessary to put a service call in to the refrigeration repair service.
- 4.6 All personnel removing samples from any refrigerator must sign them in and out. This is done by completing the ***Sample Custody Form [Attachment IV]*** which is attached to the door of each refrigerator. These completed forms are kept on file. The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the secure possession of the individual processing the samples. When the

processing is completed, the samples are returned and signed back into the appropriate storage area. It must be noted if the entire sample volume was used and that the container was discarded.

- 4.7 The water and soil walk in refrigerators are the largest refrigerators and store a large majority of the samples. There are digital min/max thermometers, which monitors the temperature 7 days a week. There are also TempAlert temperature sensors connected to each of the refrigerators for round the clock monitoring. The TempAlert sensors are set up to notify management of any exceedence of the temperature requirements.
- 4.8 A temperature maintenance record book is kept for each refrigerator. Pay close attention to these readings and watch for signs of possible problems.
- 4.9 Samples must be held for a minimum of 45 days from receipt unless specified otherwise. See SOP QS14 entitled Analytical Laboratory Waste Disposal SOP for guidance on disposal of samples.

5.0 Laboratory Information Management System (LIMS)

- 5.1 Log the sample information into the LIMS for each sample. Every attempt should be made to get every sample logged into the LIMS by the end of the day. All information entered should be clearly stated and recorded on the COC provided. After opening the main menu of the LIMS, select the 'Work Orders' tab from the 'Sample Control' drop down menu. Now click on the 'Add' button to create a new Work Order. You will see the following:
 - 5.1.1 Client: Select the client I.D. by clicking on the pull-down and choosing from the client list. This list is in alphabetical order. If the desired client is not on the list, a new client must be created by the Project Manager or DQM.
 - 5.1.2 Project: Click on 'Projects' and choose the project I.D. (**Note – correct/accurate selection is absolutely critical to the successful analysis, reporting and invoicing of all samples.**) The projects will be client specific. After the project is chosen the "project information" areas should populate. The 'Project Name,' 'Project Number,' 'TAT,' 'Client Project Manager,' 'Lab Project Manager,' and 'Comments' information should also appear. If there are no applicable project choices, a project must be created by the Project Manager or I.T. director. There are two types of projects:
 - 5.1.2.1 Internal – Empirical Laboratories projects;
 - 5.1.2.2 External – direct laboratory clients.
 - 5.1.3 Comments: This area is to be used to note any information from the Project Manager for all work orders of this project. It can also be used to list any work order specific notes; this includes but is not limited to information concerning rush turnaround, deliverables or other QC requirements, analyte concentrations, safety issues, quarantined soils, preservation or matrix problems, etc.
 - 5.1.4 Received By: Enter the name of the person who received the samples.
 - 5.1.5 Logged In By: Enter the name of the person who logged in the samples.
 - 5.1.6 Received: Enter the date and time received separated by a space and using military time. Example: 08/02/2008 08:30.
 - 5.1.7 Project/Package Date Due: After the date and time received have been entered, the date due for both of these fields will be calculated. If this information is not correct or needs to be amended later, check with the Project Manager before doing so.
 - 5.1.8 Shipping Containers: Click on the 'Coolers' button and enter the temperature and condition upon receipt. If more than one cooler was received, each cooler must be assigned a different name. For example, if these came in by dedicated courier,

enter the last four numbers of the Tracking Number as the name. After all of a cooler's information has been entered (received on ice, where custody seals present, preservation confirmed, COC/container labels agree, sample containers in-tact) click the 'Save' button. If more than one cooler was received, click the 'Add' button and repeat the process above, then click 'Done' after all the coolers' info has been saved.

5.1.9 COC Number: If an identifiable COC number is listed, record that ID here.

5.1.10 Shipped By: Enter the courier used to deliver the samples. If the samples were picked up by a lab employee or dropped off by the client/representative, enter 'Hand-Delivered.'

After these items have been completed, click 'Save,' then the 'Samples' button to continue. To begin entering information for a sample, click the 'Add' button on the bottom of the Samples screen.

5.1.11 Sample Name:

5.1.11.1 Only abbreviate if description is too long for the spaces allotted in the LIMS (must be discussed with Project Manager/Client). This information should come directly from the chain of custody. The sample ID entered into the LIMS will be the sample ID on the final report.

5.1.11.2 If no sample ID is provided, or is indistinguishable from other samples listed, contact the Project Manager to ascertain distinction in the samples. Include date as part of the description if this is the only way to differentiate the samples.

5.1.11.3 When logging in trip blanks that do not have an ID assigned by the client, list them as "Trip Blank # ____". This information should be on the containers. A log book must be kept in the sample kit room which lists all trip blanks and the date they were filled. This will ensure consistency with the descriptions for trip blanks.

5.1.12 Collection Date: Enter the date and time the sample was collected. You must use military time and separate by a space. Often the time collected is not given. Although this is a sampling requirement, this information may not be crucial unless a parameter with a short holding time or a data deliverables package is required. In the event that a sample collection time is not listed on the COC or the sample container, a default time of 00:01 can be used temporarily until client verification. Once verified, then the correct sample collection time must be input into LIMS. If the COC and sample containers do not list a collection date and time, this must be documented on the cooler receipt form and the Project Manager must be notified. All attempts should be made to get all our clients to supply this information.

5.1.13 Lab/Report Matrix: Click on pull down and select matrix. Many times it is difficult to discern the matrix if it is not specified on the COC, and log-in personnel must use their best judgment with regard to analytes/methods requested. Keep in mind that the detection limits and units on the LIMS reports are linked to the matrix. In some cases it may be necessary to ask the Section Supervisor about the matrix selection. Log-in may do a dilution test to distinguish water samples from oil samples if the COC does not clarify a sample matrix if need be.

5.1.14 Sample Type: This is used to differentiate between special types of samples (i.e. Field Duplicates, Equipment Blanks, Trip Blanks, etc.). If there is no definite way to determine that a sample should be classified as something else, then

“SAMP-Client Sample” will be selected as the sample type. Do not list a sample as anything other than a Client Sample unless noted on the COC of are instructed by the client to do so.

- 5.1.15 Container: Click on the drop down list and select the appropriate bottle type. If multiple bottles are received for the same sample, move down to the next line and select all other containers as required. Repeat this process until all containers for the sample are listed. As each container is entered, an individual number is assigned to it by the LIMS system. This number is also listed on the container labels that are printed from the LIMS, and is placed on the corresponding bottle for container tracking purposes.
- 5.1.16 pH (Container Preservative): Use this to document the pH check information taken during sample unpacking. If no preservative was used, then nothing is required in this field.
- 5.1.17 Comments: Enter any information that is applicable at the sample level.
- 5.1.18 Field Analysis: Click on field analysis tab and enter field information when provided.
- 5.1.19 Work Analyses: Select all parameters requested for the sample from this list.
 - 5.1.19.1 If the required test code is not listed, and the sample matrix is not a contributing factor, contact the Project Manager to correct the omission.
Do NOT log from “All analyses” as incorrect logins are not easily corrected.
 - 5.1.19.2 All preparation codes for analytes are entered and stored by the system independently of the test codes selected, except in the cases of Dry Weight analysis, and TCLP/SPLP preparation (tumbling). In the case of the TCLP/SPLP prep codes, these are entered alongside the other required analyses automatically by the LIMS when a TCLP/SPLP analyte is selected. As for Dry Weight, it is required for all solids testing except in the cases of TCLP/SPLP analysis, Explosives only analysis, and/or any pure product/non-soil based sample when specified by the client.
- 5.1.20 Analyses Comments: These comments should be used for any notes that only apply to that particular test code.
- 5.1.21 RTAT: If the Rush Turn-Around Time for this sample is known at the time of log-in, this information should be updated here.
- 5.1.22 Save: Once all applicable information is entered for a sample, click the save button. At this time the LIMS applies the Laboratory Sample ID to the sample. This is a five part ID code composed of the following:
 - 5.1.22.1 A 2-digit numeral of the year. Example (0811248-06).
 - 5.1.22.2 A 2-digit numeral of the month. Example (0811248-06).
 - 5.1.22.3 A 3-digit numeral of the work order number. This number reset to 001 at the beginning of each month. Example (0811248-06).
 - 5.1.22.4 A 2-digit numeral of the sample number separated by a dash. Example (0811248-06). This number is different for each sample in a work order.
 - 5.1.22.5 A container ID is also associated to every container in the form of a letter starting with “A”.
- 5.1.23 Add/Edit/Copy: Use these selections to add more samples to the work order, or to change existing information prior to label printing.
Once all the tests have been selected and all samples have been added in the work order, a work order summary and all container labels are printed. Labels

are checked for accuracy against the containers while being labeled. At this point log-in of this group of samples is complete.

- 5.2 At this point, all of the information that the primary log-in person has processed must be validated by a second person and documented on the SRC.
- 5.3 After log-in of a work order is complete and verified, the COC can then be scanned into the system and attached to the work order on the Work Order screen. The work order then must be updated to Available status so as to be seen by the analysts.

6.0 Daily Follow Up for Sample Receiving/Log In

- 6.1 Wipe out the inside of coolers and return all Empirical Laboratories coolers to the sample kit room.
- 6.2 At the end of the day organize all paperwork received and generated for the day. The following should be given to the Project Managers:
 - 6.2.1 The original chains of custody and yellow original or copy of each. The Cooler Receipt Forms will accompany the COC for the project.
 - 6.2.2 Any information (letters, regulatory limits, etc.) from a client which was received with any samples.
- 6.3 All the above information from the day will be reviewed as soon as possible.
 - 6.3.1 If any corrections or changes are required, all laboratory personnel will be notified by email distribution. E-mail notification by the Project Manager will also be sent out if a client adds or deletes any parameters, changes sample IDs, etc.
- 6.4 Sample Receiving will distribute the following to the appropriate laboratory personnel the original (white copy) chains of custody to the Reporting Department.
- 6.5 Information will be filed as follows:
 - 6.5.1 Chains of custody:
 - 6.5.1.1 Original (white copy) is returned to the customer with the final report along with the CRF.
 - 6.5.1.2 Pink copies should be retained by the sampler.
 - 6.5.2 Sample Log Change Forms
 - 6.5.2.1 Sample Log Change Forms are distributed through email to all laboratory personnel.

7.0 Miscellaneous

- 7.1 All projects which require deliverables or other QC requirements should be listed in the notes section of the LIMS.
- 7.2 If samples are received from a new client or a new job number that is not in the LIMS, a new client code must be set up. This information should be on the chain of custody or it may be necessary to contact the customer if the information is incomplete.
- 7.3 All log books used in the Sample Receiving and Sample Storage Areas are numbered. The following log books are presently maintained. All log books must be "Z"ed out. The Sample Receiving Group Leader will review the log books to check for completeness.

Log Book Description

Trip Blank Prep Log
Soil Treatment/Disposal Log
Acid Neutralization Log
Aqueous Disposal Log
Kit Room Preservation Preparation Log
Truck Maintenance Log

8.0 Sample Storage, Secure Areas and Sample Custody

- 8.1 Empirical Laboratories, LLC is located at 621 Mainstream Dr. suite 270 Nashville, TN 37228 on the first floor. This building is locked and monitored by an alarm system after normal business hours. No unauthorized personnel are permitted within the facility without a proper escort and a visitor's badge. During non business hours, all doors to the building are locked and secured by an alarm system. All front and back doors are locked and only Empirical Laboratories, LLC personnel have a key to access the building. Upon unlocking the door and entering into the laboratory, then the employee is to deactivate the alarm system using the assigned 4 digit alarm code assigned to them by Human Resources. Each employee is assigned their own designated alarm code, with no code being assigned twice. There is a buzzer at the door to Login to allow entry for sample and supply deliveries.
- 8.2 Sample extracts and digestates are stored in the following areas:
 - 8.2.1 All metals digestates are stored in the metals instrument laboratory. The transfer from the digestion analysts to the ICAP analysts is documented in the metals digestion log book.
 - 8.2.2 Non - ZHE TCLP extracts are returned to the refrigerator in which the original samples are stored. For ZHE samples, the extract is returned to the refrigerator in which the original VOC sample containers are stored.
 - 8.2.3 Extracts from medium level VOC analyses are stored in the VOC Soil Segregated Area or VOC sample freezer in the VOC Lab, depending on the associated low-level vials.
 - 8.2.4 All Organic extracts are stored in a Beverage Air side by side refrigerator in the organic extraction laboratory.
- 8.3 The generation of all sample extracts/digests and their movement through the laboratory will also be tracked on a laboratory custody sheet or in a log book. The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the secure possession of the individual processing the samples. When the processing is completed, the extracts are returned and signed back into the appropriate storage area. The metals digestates are not removed from the metals instrument laboratory.
- 8.4 After the analytical results have been reported, the original samples, sample extracts, and digestates will remain in secure storage until they are disposed of in accordance with the Waste Disposal Standard Operating Procedure (SOP QS14).

9.0 Sample Receiving Personnel Duties and Responsibilities

- 9.1 The sample receiving personnel are responsible for all duties associated to receiving, logging, subcontracting and disposing of samples. Although other laboratory personnel may assist with the duties, this is done under supervision and direction of sample receiving personnel. Sample receiving personnel are responsible for the following:
 - 9.1.1 Receive all samples for the analytical laboratory and maintain chain of custody. This includes documenting the validated time/date of receipt.
 - 9.1.2 Maintain the flow of samples through the log in process and make them available to the analysts on a timely manner. This includes prioritizing samples/projects based on turnaround requests, holding times, and second checking of primary logged sample.
 - 9.1.3 Assign the correct laboratory ID sample numbers and validate that this information is properly labeled on the containers and entered into the Laboratory Information Management System (LIMS).

- 9.1.4 Validate that every sample proceed through all steps of the log in process. This includes checking the following to determine that the sample integrity has been upheld from the time the sample is collected until it is received in the laboratory: proper containers with ample sample volume, correct preservation, sample dates/times to ensure that holding times can be met, condition of the sample containers, headspace of vials for VOC analysis, sample ID discrepancies and completeness of the chain of custody.
- 9.1.5 Communicate any information or specific requests by the client that are listed on the chain of custody, i.e., method information, detection limits, specific analytes, reporting information, turnaround information, potential hazards etc. They are also responsible for forwarding any additional information that may be received along with the samples, i.e. permit or regulatory information, letters, etc. to the laboratory managers.
- 9.1.6 The sample receiving personnel are personally responsible for continuing to uphold the sample integrity throughout the log in procedure and until the time when the samples are properly stored and disposed.
- 9.1.7 Ensure that samples are transferred into the proper storage area and that these secure areas are locked after hours.
- 9.1.8 Maintain all log books used in the section. These must be kept up to date, complete, neat and orderly.
- 9.1.9 Maintain the sample receiving and sample disposal areas in a clean, orderly and safe manner.
- 9.1.10 Follow good laboratory practices and safety procedures.
- 9.1.11 Communicate all problems, discrepancies, etc. to the Project Manager, Section Supervisor and Laboratory Director.
- 9.1.12 In situations where the client cannot be contacted, the sample receiving personnel along with the Section Supervisor must apply the best judgment on how to handle the samples or situation.
- 9.1.13 Complete all the necessary paperwork and section forms including Cooler Receipt Forms, LIMS daily print outs, Sample Receiving Custody and Disposal Form, etc. in a timely manner.
- 9.1.14 Dispose of all samples in a manner that is safe, cost efficient, timely, meets project requirements and is in accordance with hazardous waste regulations.
- 9.1.15 The sample receiving personnel are responsible for compliance of all procedures outlined in this SOP and the following SOPs.
 - 9.1.15.1 SOP QS10 Laboratory Sample Receiving, Login and Storage
 - 9.1.15.2 SOP QS11 Health and Safety Plan & Chemical Hygiene Plan
 - 9.1.15.3 SOP QS13 Field Sampling & Bottle Kit Preparation
 - 9.1.15.4 SOP QS14 Analytical Laboratory Waste Disposal

10.0 Procedure for Treatment of Soil Samples from Quarantined Areas

10.1 This summary is to explain the handling and treatment of soil samples that come from USDA quarantined areas of the United States, territories of the United States and foreign sources. This treatment is done to prevent the spread of pests to other areas. Full treatment description is addressed in SOP QS14.

10.1.1 When soil samples are ready for disposal, separate out soils that are from quarantined areas that need to be treated. Quarantined areas are from the southern United States (see attached maps), from United States territories such as Puerto Rico, and from foreign countries.

- 10.1.2 Only quarantined non-hazardous soil samples with containers that are less than three feet in depth will be treated by this procedure. Hazardous samples will have to be treated differently. A list of samples to be treated will be determined by the login supervisor.
- 10.1.3 Log the samples to be treated in the Soil Treatment Logbook as to location, date, and quantity.
- 10.1.4 Turn the oven on. Place soil samples in their containers uncovered in the oven. After oven reaches 180°C, heat samples for 2 hours. Treat container liners too. When time is up, remove soil samples with gloves or tongs and cool.
- 10.1.5 After samples have cooled, put them in the non-hazardous soil barrel for disposal.

11.0 Subcontracting Laboratory Samples

- 11.1 Sample receiving is responsible for handling all aspects of shipment of subcontracted samples. Once samples have been confirmed as sub-outs, login then notifies the Project Manager that subout samples are in house. The Project Manager then generates a purchase order number for the specific subout samples. Once the purchase order is generated by the Project Manager, then login prints out a subcontracted chain of custody from LIMS that will accompany the subout samples during transit. Then login packs up the samples into a cooler, ices them down (if necessary) to keep the samples chilled during transit, and then the cooler is shipped to the subcontracted laboratory.
- 11.2 Chain of Custody/Shipping Requirements
 - 11.2.1 When the samples are sent out, a completed sub chain of custody must be sent with the samples. Make sure to include the following information:
 - 11.2.1.1 Be specific in your analyses request. List the method number if applicable and/or any specific analytes required. This should already have been discussed with the laboratory.
 - 11.2.1.2 List the name of sub contract laboratory and the date shipped or delivered.
 - 11.2.1.3 List the Empirical Laboratories; LLC LIMS log # as the sample description on the chain of custody. Do not list the actual client name or actual project information.
 - 11.2.1.4 Record the date and time that the samples were sampled on the chain of custody.
 - 11.2.1.5 Results and invoice should be sent to the Project Manager.
 - 11.2.2 Two copies of the sub contract chain of custody should be retained. One copy should be stapled to the original chain of custody received from the client and the other should be stapled to the copy in log in.
 - 11.2.3 Make sure samples are packed well so they will not break or spill in shipment. Ice must be packed in the cooler to keep the samples cold if chilling is required.
 - 11.2.4 A P.O. must be completed and approved by the Project Manager prior to sample shipment. Sample receiving should then keep a copy of this P.O. for their records.

12.0 Attachments to QS10

- I Chain of Custody Record
- II Cooler Receipt Form
- III A) Map of Quarantined Soil Areas in the U.S./ B) Map for Fire Ants/ C) Map for Nematodes
- IV Laboratory Sample Custody Form for Walk in Refrigerator

II. EMPIRICAL LABORATORIES COOLER RECEIPT FORM

Cooler Received/Opened On: _____ @ _____ Workorder# _____
(date) (time)

1. Tracking # _____ (last 4 digits, FedEx)
Courier: ___ FED-EX UPS ROUTE WALK-IN
2. Temperature of rep. sample or temp blank when opened: _____ °C + correction factor (+ 0.2) = _____ °C
3. If Item #2 temperature is 0°C or less, was the representative sample or temp blank frozen? YES NO...NA
4. Were custody seals on outside of cooler? YES...NO...NA
If yes, how many and where: _____
5. Were the seals intact, signed, and dated correctly? YES...NO...NA
6. Were custody papers inside cooler? YES...NO...NA

I certify that I opened the cooler and answered questions 1-6 (initial/date) _____

7. Were custody seals on containers: YES NO and Intact YES...NO...NA
Were these signed and dated correctly? YES...NO...NA
8. Packing material used? Bubblewrap Plastic bag Peanuts Vermiculite Foam Insert Paper Other None
9. Cooling process: Ice Ice-pack Ice (direct contact) Dry ice Other None
10. Did all containers arrive in good condition (unbroken)? YES...NO...NA
11. Were all container labels complete (#, date, signed, pres., etc)? YES...NO...NA
12. Did all container labels and tags agree with custody papers? YES...NO...NA
13. a. Were VOA vials received? YES...NO...NA
b. Was there any observable headspace present in any VOA vial? YES...NO...NA
14. Was there a Trip Blank in this cooler? YES...NO...NA If multiple coolers, sequence # _____

I certify that I unloaded the cooler and answered questions 7-14 (initial/date) _____

15. a. On pres'd bottles, did pH test strips suggest preservation reached the correct pH level? YES...NO...NA
b. Did the bottle labels indicate that the correct preservatives were used YES...NO...NA
16. Was residual chlorine present? YES...NO...NA

I certify that I checked for chlorine and pH as per SOP and answered questions 15-16 (initial/date) _____

17. Were custody papers properly filled out (ink, signed, etc)? YES...NO...NA
18. Did you sign the custody papers in the appropriate place? YES...NO...NA
19. Were correct containers used for the analysis requested? YES...NO...NA
20. Was sufficient amount of sample sent in each container? YES...NO...NA

I certify that I entered this project into LIMS and answered questions 17-20 (initial/date) _____

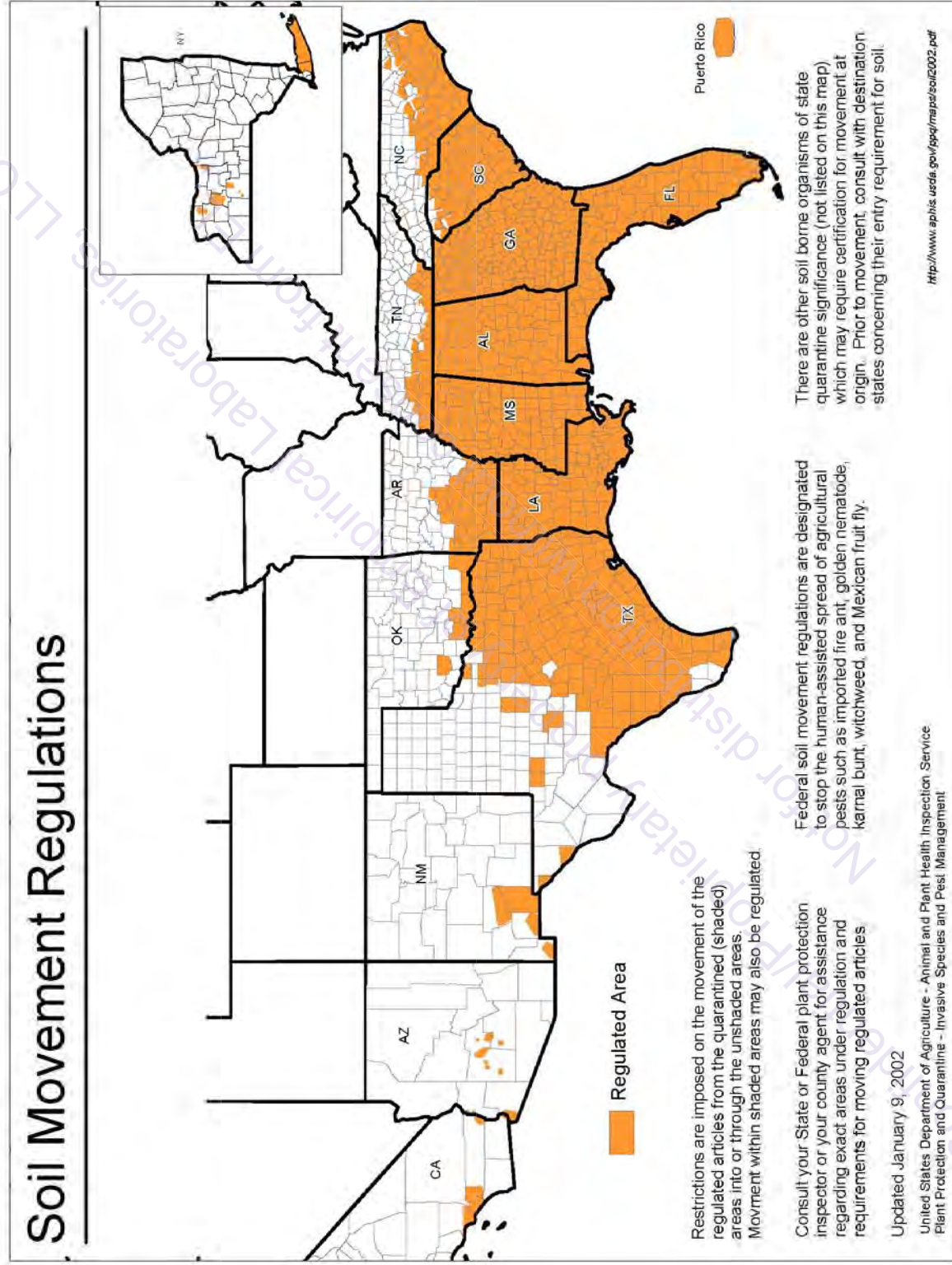
I certify that I attached a label with the unique LIMS number to each container (initial/date) _____

I certify that I have performed a second check of the LIMS information against the COC to confirm accuracy (initial/date) _____

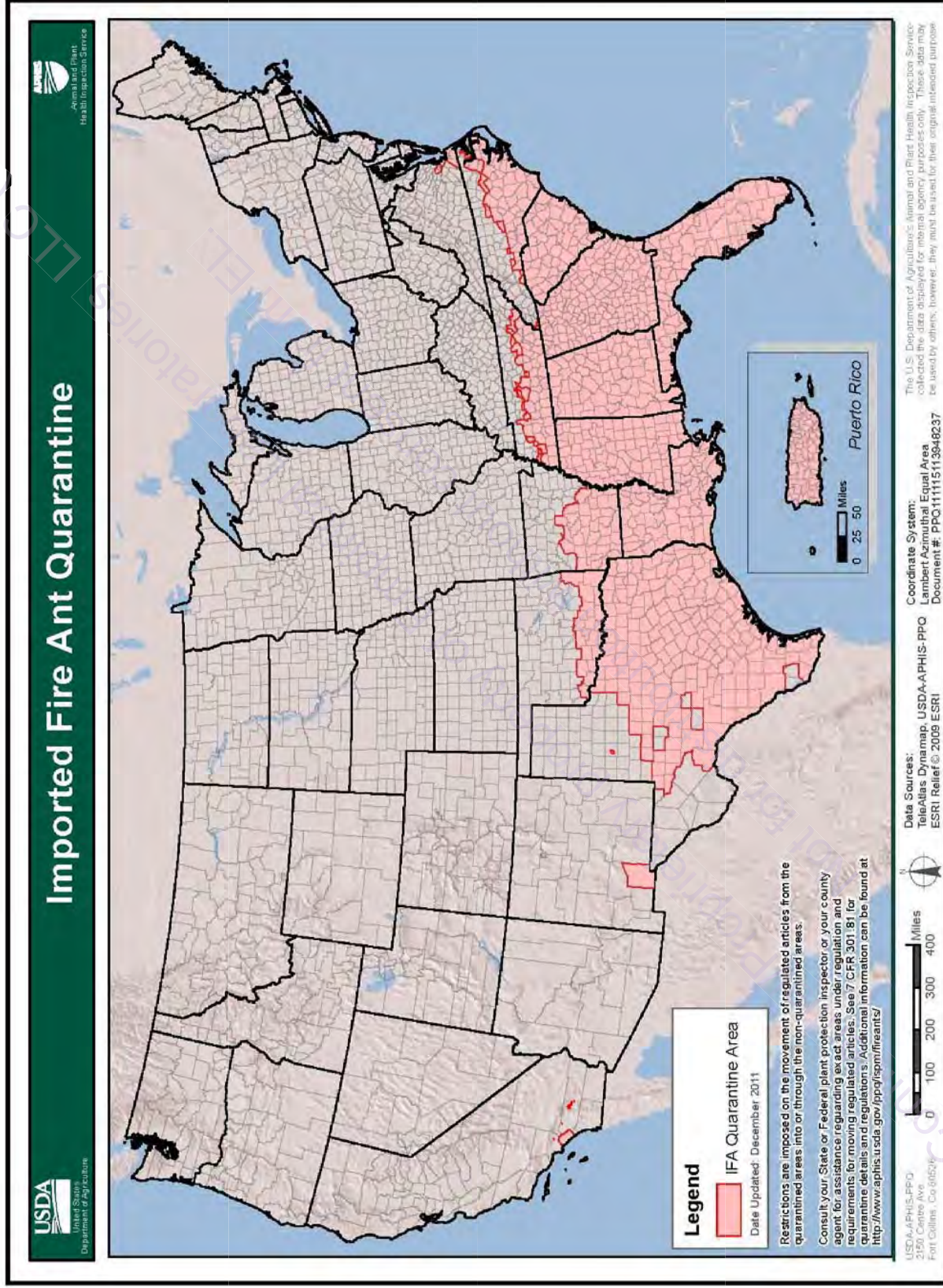
21. Were there Non-Conformance issues at login? YES...NO Was an NCR generated? YES...NO...# _____

Additional Details:

III.A) Map of Quarantined Soil Areas in the U.S.



III.B) Imported Fire Ant Quarantine Map

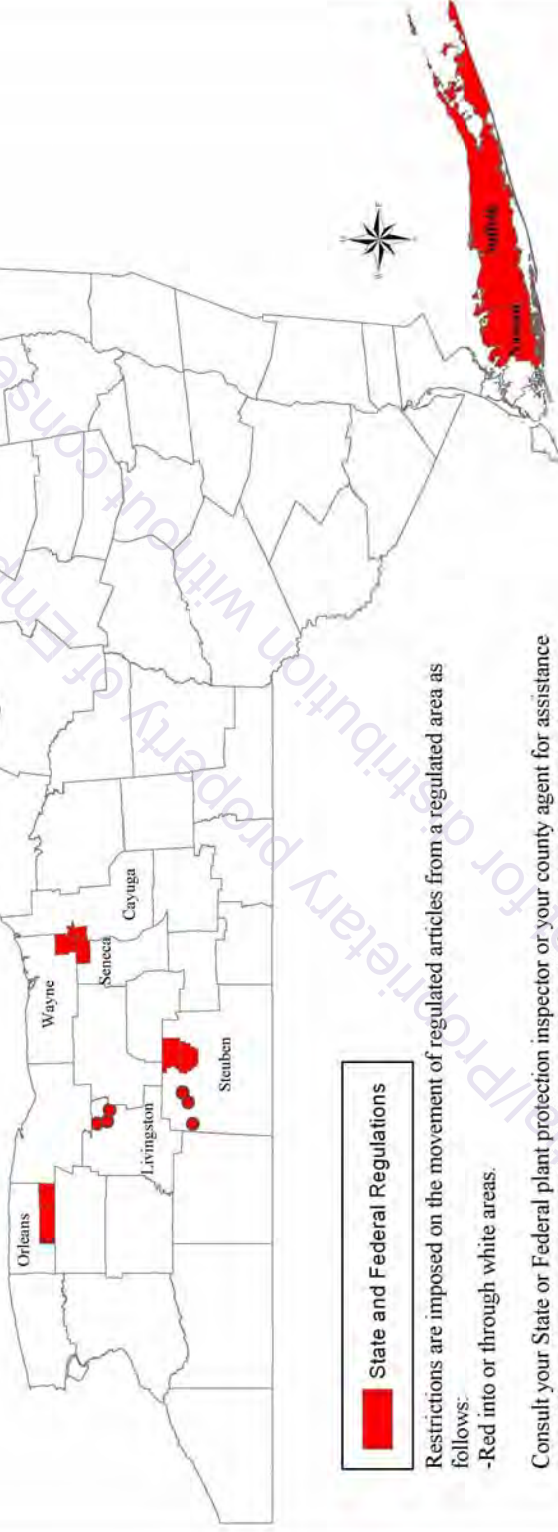


III.C) Golden Nematode Regulated Areas in New York

Golden Nematode Regulated Areas in New York

U.S. Department of Agriculture, Animal and Plant Health Inspection Service, Plant Protection and Quarantine
Cooperating with Affected States
Last updated April 2012

Counties entirely colored are
completely regulated; counties
partially colored are partially regulated.



**EMPIRICAL LABORATORIES, LLC**

WALK-IN REFRIGERATOR

[illegible]

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

QUALITY SYSTEMS: QS11

REVISION #: 16

EFFECTIVE DATE: 20140127

FIELD SAMPLING & BOTTLE KIT PREPARATION

APPROVALS:

Lab Director: _____

Date: 01/27/2014

Data Quality Manager: _____

Date: 01/27/2014

Changes Summary

Revision 16, 01/22/2014

- Section 2.3.6 – remove reference to temperature requirement North Caroling regulatory compliance samples. Updated temperature reference range from 2°C -6°C to 0°C -6°C.
- Section 2.4.3 – remove reference to Gallon Plastic (Aquatic Toxicology).
- Section 2.4.5 - remove reference to temperature requirement North Caroling regulatory compliance samples.
- Section 2.5 updated to reference requirement for all chemicals to be reagent grade and to be documented in LIMS. Added Sodium Bisulfate to list of reagents and instructions for preparing reagents.
- Section 2.6.8 updated to require tape over the FedEx label.
- Section 2.6.8.1 updated to include requirements for dangerous good stickers.
- Section 2.6.8.2 removed.
- Section 3.0 updated procedure for preparation of trip blanks then swapped with section 4.0
- Section 4.0 updated procedure for preparation of HCl preserved vials then swapped with section 3.0.
- Section 5.0 inserted for preparation of soil sampling vials for VOCs.
- TOX removed from Attachment 1
- Attachment 2 updated to indicate H₂SO₄ as the preferred acid for preservation where either H₂SO₄ or HCl are options and to update container requirements for water or soil (added “NA” where previously left empty).

Revision 15, 08/20/2012

- Reference to ISCO sampler removed.
- Attachment 2 updated to add clarification on VOA soil preservation options and handling of “ASAP” samples.
- Section 2.8 updated to remove several analyses now covered by the bottle certifications and, therefore, not required for in-house certification.

Revision 14, 12/20/10

- This SOP is an update to revision 13 dated 10/13/2010.
- Updated with changes to Attachment I on page 16 to 19 to reflect correct preservation concentration from 5mL of 1:1 H₂SO₄ acid to 2mL of 1:4 H₂SO₄ acid for the ammonia – nitrogen, COD, nitrate + nitrite, TKN, phenolics, phosphorus (total), and TOC.
- Updated with changes on page 10 section 2.5.3 for H₂SO₄ reagent concentration ratio to reflect from 1:1 ratio to 1:4
- Updated with changes to Attachment I on page 16 to 19 to reflect correct preservation concentration from 7.5mL of 1:1 H₂SO₄ acid to 2mL of 1:4 H₂SO₄ acid for the Oil & Grease, TPH, and FL-PRO.
- Updated with changes on page 14 sections 4.2.2 and 4.2.3 to reflect correct preservation amounts from 0.5mL's to 0.2mL's
- Attachment I on page 18 updated to include MEE/RSK-175 information.

Revision 13, 10/13/10

- This is an updated to revision 12 dated 09/08/2010
- This SOP was updated with changes to Attachment I on page 16 of 19 to reflect correct preservation concentration from .5mL to 0.2mL for the VOC, GRO, MEE, VPH and EDB.

Revision 12, 9/8/2010

- This is an update to revision 11 dated 10/30/2009.
- This SOP contains updated tables and references to the LIMS.

Revision 11, 10/30/2009

- Updated the SOP from the previous one into the new QS naming format and combined all the requirements for bottle kits and field sampling.
- Included the information for the qualitative certification or approved use of containers or bottles used for samples

Table of Contents

1. Field Sampling Procedures
2. Bottle Kit Preparation
3. Preparation of HCL Preserved Vials
4. Preparation of Trip Blanks
5. Preparation of VOC vials for soils

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1.0 Field Sampling Procedures

When the laboratory is employed to perform field sampling activities, the laboratory utilizes guidance from SOP-QS11, which outlines sampling guidelines for wastewater samples. A trained field sampling crew is sent to the site for sample collection and delivery of samples to the laboratory. Any relevant data such as the identification of the sampler, environmental conditions, field testing results, etc. are documented for the final interpretation of the sample results and integrity. The laboratory has a recommended Chain of Custody that is provided to the Clients; however, due to the fact that the laboratory performs only a small percentage of sampling events for the samples received, the Chain of Custody documents utilized for the sampling events could potentially be those specific to the company responsible for the sampling.

1.1 Field Sampling Overview

- 1.1.1 The laboratory shall have a sampling plan and procedures for sampling when it carries out sampling of substances, materials or products for subsequent environmental testing. The sampling plan as well as the sampling procedure shall be available at the location where sampling is undertaken. The sampling process shall address the factors to be controlled to ensure the validity of the environmental test results. Where sampling (as in obtaining sample aliquots from a submitted sample) is carried out as part of the test method, the laboratory shall use documented procedures and appropriate techniques to obtain representative subsamples.
- 1.1.2 Where the client requires deviations, additions or exclusions from the documented sampling procedure, these shall be recorded in detail with the appropriate sampling data and shall be included in all documents containing environmental test results, and shall be communicated to the appropriate personnel.
- 1.1.3 The laboratory shall have procedures for recording relevant data and operations relating to sampling that forms part of the environmental testing that is undertaken. These records shall include the sampling procedure used, the identification of the sampler, environmental conditions (if relevant) and diagrams or other equivalent means to identify the sampling location as necessary and, if appropriate, the statistics the sampling procedures are based upon.

1.2 Field Sample Collection

- 1.2.1 The field sampler is responsible for taking the necessary bottleware and personal protection wear (gloves/safety glasses) into the field in preparation for collecting samples. The appropriate sample ID's and/or parameter containers should be labeled so that once the field sampler has arrived to the sampling site, he/she would know which bottles to use for which requested parameters (I.E. which containers are grab versus composite, etc.). Once the field sampler arrives at the sampling site, a grab or composite (and/or both) sample will be collected. This is entirely dependent on client specifications.
- 1.2.2 For grab samples, using the appropriate parameter container, the sampler will "scoop" an aliquot amount filling the container of sample volume.
- 1.2.3 In some instances, the client may request for a field pH to be taken for wastewater samples on-site during a sampling event. This is done by the field sampler using a field pH meter. The field sampler takes the field pH meter into the field along with cohesive probe, buffer solutions, and DI water. Once a water sample is

collected, the field sampler will manually calibrate the pH meter by doing the following:

- 1.2.3.1 First, a buffer solution with a pH of 7 will be used to calibrate the pH meter. The probe is first immersed into DI water to cleanse the probe then immersed into the pH 7 solution and then the field sampler will wait until the pH meter equilibrates what pH the probe is reading. If the pH is above or below 7, the sampler will then adjust the calibration screw using the included flat head screwdriver to adjust the calibration screw until the pH meter indicates a pH of 7.
- 1.2.3.2 Buffer solutions that have a pH of 4 and 10 will be used to calibrate the slope of the pH meter. The sampler then immerses the probe into both the pH 4 buffer solution and the pH 10 buffer solution and adjusts the slope calibration screw using a flat head screwdriver to turn and adjust the calibration screw until the pH meter reads the correct corresponding buffer solution pH reading (i.e. if dipping into the pH 4 buffer solution the pH of the meter should read 4 and pH 10 solution should read a pH of 10 on the pH meter). *NOTE: The pH probe MUST be immersed into the DI water solution and rinsed off between each pH buffer solution calibration check. When checking the pH of different samples, the DI water solution must be used to rinse the pH probe after the reading of each individual samples before proceeding to the next.*
- 1.2.3.3 After all calibration is complete; it is ready to be used for actual samples collected during the sampling event. Insert the probe into the appropriate sample container and once the pH meter has the same exact reading for approximately 10 seconds, this is the final pH of the sample that will be recorded on the chain of custody.

Once all samples have been collected in the field and all sampling tools have been gathered, samples are then transported back to the laboratory and the appropriate client sampling event chain of custody (COC) is filled out by the field sampler. This COC must include client name, sampling site/event, sample ID's, sample dates and times collected, if the samples are grab or composite, parameters requested, and the field samplers printed name or signature.

2.0 Bottle Kit Preparation

- 2.1 This SOP is for the preparation of sample kits which are prepared by Empirical Laboratories, LLC laboratory personnel and sent to our customers for collecting samples. Sample kits can be prepared for all laboratory projects but the contents of the kit will vary depending on the sample matrix (i.e., drinking, surface, ground, and waste waters, sediments, soils, sludges, wipes, products, and others) and project specific requirements (i.e., compliance monitoring, state regulations, QC requirements, etc.). Our goals when preparing kits are:

- 2.1.1 To provide the customer with the proper containers and chemical preservatives as specified by the USEPA, chain of custody record, temperature blank, etc. in an

effort to uphold sample integrity and ensure that the data obtained from the analyses is valid.

- 2.1.2 To make the collection of the samples easier for our customers by providing them with containers which are pre-labeled, pre-preserved and organized by sample locations, supplying project specific instructions and sampling information (i.e., VOA sampling, QA/QC issues, regulatory requirements, etc.).
- 2.1.3 To make the return of the samples to the laboratory and sample log in process as easy as possible and minimize problems.
- 2.1.4 To continue to evaluate the needs of our customers and continually fine tune our process to provide them with the information and supplies they need for collecting samples. Also to be aware of the most cost effective approaches without sacrificing quality.
- 2.1.5 To ensure that samples are collected with adequate sample volume including additional volume if retesting is necessary and for QA/QC requirements, i.e., blanks, matrix spikes/ matrix spike duplicates (MS/MSD), etc.
- 2.2 Summary:
 - 2.2.1 Sample kits containing the proper sample containers, preservatives (if necessary), chain of custody record, sampling instructions (project specific) and other project specific supplies/information are prepared by laboratory personnel and sent to the client/sampling site. These supplies are normally packed in a sample cooler along with packing material to prevent breakage to the site and in return to the laboratory.
- 2.3 Interferences:
 - 2.3.1 The following items must be examined when preparing sample kits as they may cause interferences during analyses. Although the analyses can still be performed it may be viewed as an "out of compliance" situation by regulatory agencies.
 - 2.3.2 Type of Container: Samples must be collected in the proper type of container as specified by the USEPA or DOD and project specific requirements. This includes glass vs. plastic, Teflon lined lids vs. plastic lined, septum lids to prevent headspace, etc.
 - 2.3.3 Preservation Requirements: The proper chemical preservative must be used and the samples maintained at the proper temperature.
 - 2.3.4 Adherence to USEPA Holding Times: Samples should be shipped overnight or delivered immediately to the laboratory especially for parameters with limited holding times (7 days or less). If analyses are required which have a 48 hr. or less holding time this information should be noted on the Sample Kit Form.
 - 2.3.5 Chemical preservatives (acids and bases): May cause interferences if the stock solutions have become contaminated or if they are not the proper analytical grade.
 - 2.3.6 Temperature preservation: Samples should be put on ice immediately after they are collected and should be maintained at 0°C -6 °C as part of the required preservation. The only exceptions to this are:
 - 2.3.6.1 Water samples for Fluoride, Bromide and Chloride
 - 2.3.6.2 Water samples for all Metals except Chrome 6+ and Mercury that have been preserved with HNO₃ to a pH of <2.
 - 2.3.6.3 Waste or product samples and samples from "other" matrices such as glass, wood, etc.

Attachment 2 displays the recommended sample volumes, sample containers, preservations, and holding times for collection of ground water, surface water, and soil/sediment samples.

2.4 Equipment/Supplies:

- 2.4.1 Cooler: small, medium, or large size depending on the size of the sample kit. Any cooler that is cracked, broken beyond repair, filthy, etc. must be discarded.
- 2.4.2 Containers: the type of container is determined by required parameters and project specific requirements. The following issues must be examined to determine what type of container is required:
 - 2.4.2.1 Volume of sample required for a particular analysis (always plan for extra in case problems occur). If specific QC [MS/MSD, Lab duplicate, etc.] is required, extra volume must be collected.
 - 2.4.2.2 Type of preservative required for a particular analysis. Several parameters may be combined in the same container if they require the same chemical preservatives.
 - 2.4.2.3 Matrix of the sample, i.e., non aqueous vs. aqueous
 - 2.4.2.4 Container classification:
 - 2.4.2.4.1 New
 - 2.4.2.4.2 Precleaned* without certification
 - 2.4.2.4.3 Precleaned* with certification

**Cleaned and certified in accordance with the USEPA manual "Specifications and Guidance for Contaminant-Free Sample Containers."*
- 2.4.3 Various sizes of glass and plastic containers are kept in stock for use in sample kits. The following list are kept in stock and most commonly used, although specific projects may require a size or type of container different from these listed.
 - 1 liter plastic
 - 500 mL. plastic
 - 250 mL. plastic
 - 120 mL Plastic
 - Cubetainers (Houses DI water for field blanks)
 - 1 liter glass amber
 - 40mL VOA vials
 - 4oz. amber glass soil jars
- 2.4.4 Packing Material - Foam sleeves, bubble wrap, and vermiculite are most commonly used.
- 2.4.5 Bagged ice – This is much more efficient in cooling and maintaining the sample between 0°C-6 °C
- 2.4.6 Chain of Custody - An Empirical Laboratories, LLC chain of custody record must be sent in each cooler. Additional copies should be sent for back up purposes. The client may choose to complete their own chain of custody.
- 2.4.7 Custody Seals - make sure enough as they may be required per each container or just on the outside of each cooler
- 2.4.8 Labels:

- 2.4.8.1 Zebra printer white labels or generic manufacturer labels for listing sample information.
- 2.4.8.2 Preservative Stickers - used when a sample container has had chemical preservative added.
- 2.4.8.3 Other labels – “Contains Wet Ice” and “Fragile”. Cooler return labels are used for shipment of collected samples back to the laboratory.
- 2.4.9 Garbage bags - used to line the coolers for return shipment to help prevent leakage from melting ice or in a case a container spills.
- 2.4.10 Ziploc bags - used to keep chain of custody and other paperwork from getting wet.
- 2.5 Reagents: The laboratory’s LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. **All reagents shall be made from ACS reagent grade chemicals and documented within the LIMS.** Reagents are purchased from Fisher, EMD, TN Reagents or an approved alternate. Expiration date is 365 days unless otherwise noted.
- 2.5.1 1:5 Nitric Acid (HNO_3) (*concentrated nitric acid cannot be shipped by ground or air by any commercial shipping company.*) - slowly add 200mL of HNO_3 to ~500mL of reagent water in a 1000mL container then bring to final volume of 1000mL with DI water. This mixture will get very warm. Allow to cool before use.
- 2.5.2 1:1 Hydrochloric Acid (HCl) – slowly add 250mL of HCl to ~125mL of reagent water in a 500mL container then bring to final volume of 500mL with DI water. This mixture will get very warm. Allow to cool before use. Note: Designated HCl must be used when preparing for VOA 40mL vial preparation. Prepare in VOA lab using fresh GC/MS VOA lab DI water and set expiration date to 90 days from preparation.
- 2.5.3 1:4 Sulfuric Acid (H_2SO_4) - slowly add 125mL of H_2SO_4 to ~250mL of reagent water in a 500mL container then bring to final volume of 500mL with DI water. This mixture will get very warm. Allow to cool before use.
- 2.5.4 6 Normal (6N) Sodium Hydroxide (NaOH) – weigh 240g NaOH and add to ~250ml DI water in a 1000mL container. Swirl to dissolve then bring to final volume of 1000mL with DI water.
- 2.5.5 1 Normal (1N) Zinc Acetate (ZnAc) – weigh 55g ZnAc and add to 125mL DI water in a 250mL container. Swirl to dissolve then bring to final volume of 250mL with DI water.
- 2.5.6 Sodium Thiosulfate ($\text{Na}_2\text{O}_3\text{S}_2$), used as preservation for coliform analyses. We use sterilized containers which are received from the manufacturer with the Sodium Thiosulfate already in the container. These may also be used for organic analyses, preserving drinking water VOC’s, or other samples that contain residual chlorine.
- 2.5.7 Ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$) is used to remove chlorine from cyanide samples. Approximately 1 to 2 grams will be added for each cyanide sample container (if they contain residual chlorine due to being a chlorinated effluent).
- 2.5.8 Analyte Free Water (DI), sent to the site for rinsate (equipment) and/or field blanks. The water must be taken from the same water source as used in the laboratory.

- 2.5.9 Sodium Bisulfate (NaHSO₄, ACS Grade, Granular), Each lot of granular reagent is to be tested before use by adding 1g reagent with 5mL DI water in 40mL VOA vial and providing to receiving for logging with ID NaHSO₄/Lot#_____ then giving to VOA analyst for analysis. To make a 20% solution, weigh 100g NaHSO₄ and add to 250mL DI water in a 500mL container. Swirl to dissolve then bring to final volume of 500mL with DI water. Expiration date is 14 days from preparation.

NOTE: See corresponding bottle containers and preservation amounts chart (attachment I) in this SOP for further details.

2.6 Sample Kit Procedure

The laboratory project manager prepares a "sample kit form" which details the type and number of samples, type and number of required containers, type of preservation required, project specific requirements, etc. (methods, detection limits, QA/QC etc.). This order will also list the client, address, method of shipment, date required, etc.

- 2.6.1 A label is placed on each sample container with lines for the following information:
- 2.6.1.1 Client name (optional)
 - 2.6.1.2 Project Name (if applicable)
 - 2.6.1.3 Sample ID (may be filled in by the person preparing the kit or may be left blank for the sampler to complete)
 - 2.6.1.4 Date and Time Collected (left blank for the sampler to complete)
 - 2.6.1.5 Parameters (normally filled in by the kit preparer)
 - 2.6.1.6 Parameter Methods (normally filled in by the kit preparer)
 - 2.6.1.7 Preservative (normally filled in by the kit preparer)
 - 2.6.1.8 Terracore Vial Weight (If applicable)
- 2.6.2 Labels can be prepared to the client's specifications in most cases. In some instances, we do not supply labels (client request) or do not attach them to the bottles; they are placed inside the sample kit.
- 2.6.3 If chemical preservatives are added to the sample bottles, a preservation label listing the type of preservative must be placed on the container. For safety reasons, and in order to minimize contamination, chemical preservations are in general added to the sample bottles prior to transporting to the field.
- 2.6.4 Normally sample containers are organized by sample locations and then packed into coolers. For kits requiring a large # of parameters, pack one sample location per cooler. For those requiring a smaller list of parameters, several sites may be packed into one cooler. There may be project specific requests such as: putting all VOC vials into one cooler, packing one cooler per each day, etc. The sample containers should be packed in the cooler so that there is no movement during shipment.
- 2.6.5 Sample bottles must be packed in a cooler with foam sleeves, bubble wrap or other packing material. Make sure that enough packing material is included for return shipment to the laboratory. This will help prevent the bottles from breaking during shipment to and from the laboratory.
- 2.6.6 A plastic bottle filled with water (tap water is fine) must be placed in every cooler. Label this bottle as a "temperature control blank". This bottle will be used to

check the temperature of the samples upon receipt in the laboratory, and used for transit monitoring to ensure that the samples are properly chilled.

- 2.6.7 Every cooler must also contain a plastic Ziploc bag with the following:
 - 2.6.7.1 Chain of custodies. This form is provided to allow the sampling personnel to enter all the necessary sample information and document official release of the samples to the laboratory.
 - 2.6.7.2 A copy of the sample kit form. This will help the sampling personnel to know how many and what kinds of containers to fill.
 - 2.6.7.3 Cooler return labels that can be used by the client sampling personnel, which provides them with a return address to our laboratory to attach to the cooler housing samples to be sent to the laboratory for analysis.
 - 2.6.7.4 Custody seals, which are filled out by the client and attached to the coolers to ensure that the coolers shipped are sealed and have not been opened during transit.
- 2.6.8 We normally ship sample kits to clients by Federal Express. Make sure the FedEx label is completely covered by shipping tape when placing on the cooler. If chemical preservatives are used, the kits must be shipped in accordance with the courier's regulations.
 - 2.6.8.1 Federal Express (Fed Ex) - Because the amount of preservative used is such a small amount we are allowed to ship in accordance with limited quantities regulations. Dangerous goods stickers are required for any coolers (air or ground shipment) containing Zinc Acetate (ZnAc), Sodium Hydroxide (NaOH), Methanol (MeOH) and/or Sodium Bisulfate (NaHSO₄).
 - 2.6.8.2 Kits may also be picked up by the customer and driven to the site or delivered by Empirical Laboratories, LLC personnel.
- 2.6.9 The cooler lid must be taped shut so that it will not open during shipment. It is best if the tape is placed around the entire cooler being certain that it overlaps. This will prevent the tape from coming off during shipment. If a cooler comes open during shipment it may be returned to the laboratory, may spill the contents, or slow up shipment to the customer.
- 2.6.10 A record of all sample kits prepared is kept in the sample kit room. The client, date, method shipped, number of coolers, and any other miscellaneous information (tracking numbers, certification paperwork, etc.). This information must be kept up to date.

2.7 Quality Control

- 2.7.1 Trip Blanks can be sent with sample kits for projects that require VOC analyses per client request. These trip blanks are prepared away from areas where solvents are used or stored and as close to the time of shipment as possible in order to keep storage time to a minimum.
- 2.7.2 Duplicate field samples can be taken if requested by the client. Additional containers must be included in the sample kits if this is required.
- 2.7.3 Equipment (rinsate) blanks and/or field blanks can be taken if requested by the client. Additional containers must be included in the sample kits for this.
- 2.7.4 Matrix spikes and matrix spike duplicates can also be performed if requested by the client. This type of QC may also be done for internal use on projects with

large number of samples. Additional containers must be included in the sample kits for this.

- 2.7.5 If projects require containers that have been certified as contaminant-free, the certification papers are maintained on file.
- 2.7.6 All sample containers and chemical preservatives are stored in an area isolated from any areas where solvents are used and stored.
- 2.7.7 Complete traceability of the preservatives used to pre-preserve the sample containers and to preserve samples is required. A bound notebook must be used to trace this information and must include the following: lot #, type of preservative, date prepared, amount and analyst name. This information must also be labeled on each container, re-pipetter, etc. that the preservative is stored in. Each lot of HNO₃ used for Metals preservation should be tested prior to using them for preservation. These analyses are kept on file by the Inorganics Manager.
- 2.7.8 Containers that have been pre-cleaned and certified in accordance with the DOD or USEPA manual ("Specifications and Guidance for Contaminant-Free Sample Containers" QSWER Directive # 9240.0-05A Dec. 92) are used.

2.8 In House Bottle Lot Certifications

Due to regulatory requirements, EL assesses the cleanliness of containers by performing the appropriate analysis on one container per lot used. The analytical assessment is performed to half of the laboratory's Limit of Quantitation (or LOQ). When internal analysis is performed on containers, the bottle is logged into LIMS and provided to the analytical group for analysis. This allows for appropriate tracking of the analysis by assigning an appropriate LIMS Work Order #. In some cases, the vendor is able to provide the appropriate analytical certifications for the lot of containers used. In which case, the laboratory may not perform additional testing internally. The certifications provided by the bottle vendors are filed by the custody section and available for review.

- 2.8.1 Certified clean sample containers (for DOD clients) for the following inorganic chemistry parameters: sulfide, perchlorate, total organic carbon, anions (chloride, sulfate), ICP metals (TAL list), and ICP metals low level are certified in house. The certifications for the parameters listed above are tested in house and the results are kept on file.
- 2.8.2 Certified clean sample containers (for DOD clients) must be used for the following organic parameters which are certified in house: herbicides, semi-volatiles, explosives, GRO, DRO, and low level PAH. These are certified in house for both waters (using unpreserved 1L amber glass bottles) and soils (using 4oz amber glass soil jars) and the results are kept on file.
- 2.8.3 New certified clean sample container 40mL vials (for DOD clients) must be used for the following parameters which are certified in house: volatile organic compounds, MEE (methane, ethane, and ethane), GRO, and method 8011 (EDB and DBCP). These results are kept on file.

2.9 Miscellaneous Information

- 2.9.1 When preparing sample kits for collecting non aqueous samples for both Inorganic and Organic analyses, separate containers should be sent. There may be some situation where we may not receive separate containers upon return to the laboratory (i.e., not enough sample volume, containers received that we did not prepare, breakage, etc.).

- 2.9.2 For VOC analyses on aqueous samples, three 40 mL vials should be sent for each sample. Normally additional volume from the three vials is required for MS/MSD.
- 2.9.3 For Oil and Grease only one container should be sent in the sample kit. It may be necessary on some projects to send two containers.
- 2.9.4 For Semi-Volatile (BNA) Pesticide and/or PCB, and Herbicide analyses two containers should be sent. Additional containers must be sent for MS/MSD. When multiple 1L parameters are being submitted, for instance BNAs, Pest/PCBs and herbicides, a reduced number of backup containers may be shipped.
- 2.9.5 Normally the number of bottles sent other than VOCs depends on the client's kit requests.
- 2.9.6 Note that in some instances, it may be necessary to have sample containers shipped directly from the supply vendor to the sampling site. Additional supplies such as preservatives, organic free water for the rinsate blanks, etc. may still be supplied by the laboratory and will follow all procedures of this SOP.
- 2.9.7 When preparing trip blanks for VOCs, two vials are normally sent. Label and number each set of vials. Record all trip blanks including their number, date prepared, project/client name, lot number, preservation, and analyst initials in a designated log book which is kept in the sample kit room.

3.0 Preparation of HCL Preserved Vials

3.1 Equipment/Apparatus:

- 3.1.1 1:1 Hydrochloric Acid (Designated for VOA preservation).
- 3.1.2 Unpreserved, certified 40mL VOA vials.
- 3.1.3 Hydrochloric Acid Dispenser (cleaned and filled with fresh HCl, adjustable in 0.05mL increments).
- 3.1.4 Appropriate lab protection attire (safety gloves, glasses, lab coat).

3.2 Procedure:

- 3.2.1 Take a box of unpreserved, certified VOA vials to the preparation cubicle (preferably all of the same lot number from the manufacturer).
- 3.2.2 Make sure that the HCL dispenser is set at an aliquot of 0.2 mL's. Make sure that you use a dispenser that uses increments of 0.05mL.
- 3.2.3 Remove the cap from the VOA vial and dispense 0.2mL's of HCL acid into the vial making sure not to touch the tip of the dispenser to the vial.
- 3.2.4 Immediately replace the cap.
- 3.2.5 Fill a box of 72 vials (or more, as needed) in the same manner.

4.0 Preparation of Trip Blanks Procedure - Because of the ease of contaminating trip blank water with common laboratory contaminants, a procedure must be in place to ensure that the trip blank water is clean before sending it to the field.

4.1 Equipment/Apparatus:

- 4.1.1 The DI water system located in the GC/MS VOA laboratory.
- 4.1.2 HCL preserved VOA vials – preserved as in section 3.0 above or purchased certified from a reputable vendor.
- 4.1.3 Appropriate lab protection attire (safety gloves, glasses, lab coat).
- 4.1.4 VOA trip blank log book – stored in sample receiving.

4.2 Procedure:

- 4.2.1 Take a box of certified, HCl preserved VOA vials to the GC/MS VOA Laboratory, preferably all one lot # from the manufacturer or one lot # preserved per section 3.0 above.
- 4.2.2 Remove the cap from the VOA vial and fill the vial with water from the water system so that there is a convex bubble on the top of the vial of water.
- 4.2.3 Place the cap on quickly, making sure there is no air-space in the vial.
- 4.2.4 Check for bubbles in the vial by rotating 180° while looking for bubbles.
- 4.2.5 Fill a box of 72 vials (or more, as needed) in the same manner.
- 4.2.6 Label the box of vials with the Lot # and Date that they were prepared.
- 4.2.7 Record this lot # and date in the designated VOA trip blank logbook.
- 4.2.8 Log a vial into LIMS for testing with lot number and date of preparation in the sample ID. Add the workorder number to the box of trip blanks and initial/date when results are received and acceptable.
- 4.2.9 The fully labeled box of trip blanks is stored in the preparation cubicle until completely used.

5.0 Preparation of VOC vials for soils.

5.1 Equipment and apparatus

- 5.1.1 DI water – dispenser from GC/MS VOA lab - to be filled fresh before each use from the DI water system in the GC/MS VOA lab.
- 5.1.2 NaHSO₄ (ACS Grade, Granular), 20% solution prepared within the last 14 days, in dispenser adjustable in 0.05mL increments.
- 5.1.3 Methanol (Purge and Trap grade) – dispenser from GC/MS VOA lab.
- 5.1.4 Stirbar, ESS No. 800370137 or equivalent
- 5.1.5 VOA soil vial preparation log book (stored in kit preparation area)

5.2 Procedure

- 5.2.1 Take a box of unpreserved, certified VOA vials to the preparation cubicle (preferably all of the same lot number from the manufacturer).
- 5.2.2 Make sure dispenser is set to 5.0mL
- 5.2.3 Two low-level vials and one methanol vial are routinely provided.
- 5.2.4 It is imperative that these vials always be prepared in the GC/MS VOA lab or the designated preparation cubicle.
- 5.2.5 Add 5mL of appropriate reagent (DI water, NaHSO₄ or MeOH) and stir-bar (if DI water or NaHSO₄ vial).
- 5.2.6 Complete label with preservative and assigned lot number then affix to the vial.
- 5.2.7 After all reagents, stir bars and labels have been added, weigh vial on an appropriately calibrated balance which reads to 2 decimal places (0.00g).
- 5.2.8 Record this weight (tare weight) in the lower right side of the vial label and in the VOA soil vial preparation log book.

Attachments to SOP-QS11:

- 1) Corresponding Bottle Containers and Preservation Amount**
- 2) Method Preservation Summary**

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Attachment 1: Corresponding Bottle Containers and Preservation Amounts

Parameter	Bottle Type (Glass or Plastic)	Bottle Size	Preservation	How Much Preservation (mLs)
Ammonia – Nitrogen	Bel Art Plastic	250mL	H2SO4	2mL of 1:4 concentrated H2SO4 Acid
COD	Bel Art Plastic	250mL	H2SO4	2mL of 1:4 concentrated H2SO4 Acid
Cyanide (Total, Amenable)	Bel Art Plastic	250mL	NaOH	2mL of 120g NaOH:500mL of H2O (6N)
Hardness	Bel Art Plastic	250mL	HNO3	2mL of 1:5 concentrated HNO3 Acid
Nitrate+Nitrite, as N	Bel Art Plastic	250mL	H2SO4	2mL of 1:4 concentrated H2SO4 Acid
Nitrogen, Total Kjeldahl (TKN)	Bel Art Plastic	250mL	H2SO4	2mL of 1:4 concentrated H2SO4 Acid
Oil and Grease (Gravimetric)	Glass Amber	1000mL	H2SO4 (preferred) or HCL	2mL of 1:4 concentrated H2SO4 Acid or 2.5mL of 1:1 concentrated HCL Acid
Petroleum Hydrocarbons, Total (TPH)	Glass Amber	1000mL	H2SO4 (preferred) or HCL	2mL of 1:4 concentrated H2SO4 Acid or 2.5mL of 1:1 concentrated HCL Acid
Petroleum Hydrocarbons, Extractable (EPH)	Glass Amber	1000mL	H2SO4 (preferred) or HCL	2mL of 1:4 concentrated H2SO4 Acid or 2.5mL of 1:1 concentrated HCL Acid
Diesel Range Organics (DRO)	Glass Amber	1000mL	H2SO4 (preferred) or HCL	2mL of 1:4 concentrated H2SO4 Acid or 2.5mL of 1:1 concentrated HCL Acid
Phenolics, Total	Glass Amber	1000mL	H2SO4	2mL of 1:4 concentrated H2SO4 Acid
Phosphorus, Total	Bel Art Plastic	250mL	H2SO4	2mL of 1:4 concentrated H2SO4 Acid
Sulfide, Total	Bel Art Plastic	250mL	ZnAc/NaOH	2mL of 55g ZnAc:250mL of H2O (1N) + 1.5mL of 120g NaOH:500mL of H2O (6N)
Total Organic Carbon (TOC)	Bel Art Plastic	250mL	H2SO4 (preferred) or HCL	2mL of 1:4 concentrated H2SO4 Acid or 1mL of 1:1 concentrated HCL Acid
FL-PRO	Glass Amber	1000mL	H2SO4	2mL of 1:4 concentrated H2SO4 Acid
Total & Dissolved Metals (Including Hg) (Dissolved must be field filtered prior to preserving)	Bel Art Plastic	250mL	HNO3	2mL of 1:5 concentrated HNO3 Acid
Volatile Organic Compounds (VOC) Gasoline Range Organics (GRO) Methane, Ethane, Ethene (MEE) Volatile Petroleum Hydrocarbons (VPH) EDB/DBCP	Glass Vial	40mL	HCL	0.2mL of 1:1 concentrated HCL Acid

<u>Attachment 2 – METHOD PRESERVATION SUMMARY</u>		<u>CONTAINERS</u>		<u>PRESERVATION</u>	<u>HT (days)</u>
<u>ANALYSIS</u>	<u>METHOD</u>	<u>WATER</u>	<u>SOIL</u>	~soils get NO preservation~¹	<u>Water / Soil</u>
<u>General Chemistry Methods</u>					
Acidity	SM2310B	250mL / Plastic	NA	none / cool to 4°C	14 / NA
Alkalinity	SM2320B	250mL / Plastic	NA	none / cool to 4°C	14 / NA
Ammonia-N	SM4500NH3BG	250mL / Plastic	4oz. glass jar	H2SO4 / cool to 4°C	28 / 28
Anions (excluding below)	9056A / 300.0	250mL / Plastic	4oz. glass jar	none / cool to 4°C	28 / 28
Anions (Nitrate-N / Nitrite-N)	9056A / 300.0	250mL / Plastic	4oz. glass jar	none / cool to 4°C	48 hrs / 28
BOD	SM5210B	1L / Plastic	NA	none / cool to 4°C	48 hrs / NA
COD	410.4	250mL / Plastic	NA	H2SO4 / cool to 4°C	28 / NA
Coliform, Fecal & Total	9131, 9132	250mL / Plastic	NA	Sodium Thiosulfate / cool to 4°C	6 hrs / NA
Conductance, Spec.	9050A	250mL / Plastic	NA	none / cool to 4°C	28 / NA
Corrosivity (pH)	9040B / C, 9045C / D	250mL / Plastic	4oz. glass jar	none / cool to 4°C	ASAP ² / 28
Cyanide, Total	9012A / B / SM4500-CNG	250mL / Plastic	4oz. glass jar	NaOH / cool to 4°C	14 / 14
Hardness	6010B / C / 200.7	250mL / Plastic	NA	HNO3 / cool to 4°C	180 / NA
Hydrogen Ion, pH	9040B / C, 9045C / D	250mL / Plastic	4oz. glass jar	none / cool to 4°C	ASAP ² / 28
Nitrate+Nitrite-N	353.2	250mL / Plastic	4oz. glass jar	H2SO4 / cool to 4°C	28 / 28
Oil and Grease	1664A	1L / Glass	4oz. glass jar	H2SO4 / cool to 4°C	28 / 28
Oil and Grease	9071B	NA	4oz. glass jar	cool to 4°C	NA / 28
Oxygen, Diss. Probe	SM4500G	250mL / Glass	NA	none / cool to 4°C	ASAP ² / NA
Paint Filter	9095B	250mL / Plastic	4oz. glass jar	none / cool to 4°C	28 / 28
Perchlorate	6850 or 314	250mL / Plastic	4oz. glass jar	none / cool to 4°C	28 / 28
Phenolics	420.2	1L / Glass	4oz. glass jar	H2SO4 / cool to 4°C	28 / 28
Phosphorate, Ortho	SM4500PE	250mL / Plastic	NA	none / cool to 4°C	48 hrs / NA
Phosphorus, Total	SM4500PBSE	250mL / Plastic	4oz. glass jar	H2SO4 / cool to 4°C	28 / NA
Settleable Solids	SM2540F	1L / Plastic	NA	none / cool to 4°C	48 hrs / NA
1 – Volatile soils can be field sampled / preserved in 40mL VOA vials with 5g to 5ml sodium bisulfate solution or methanol to provide a 14 day holding time when cooled to 4°C.					
2 - Field tests - to be performed within 15-minutes, usually samples are out of hold when received, upon receipt test as soon as possible.					

Attachment 2 – METHOD PRESERVATION SUMMARY		CONTAINERS		PRESERVATION	HT (days)
ANALYSIS	METHOD	WATER	SOIL	~soils get NO preservation~¹	Water / Soil
General Chemistry Methods (contd.)					
Sulfide	SM4500S-2 CF / 9034	250mL / Plastic	4oz. glass jar	ZnAC / NaOH / cool to 4°C	7 / 7
TDS	SM2540C	500mL / Plastic	NA	none / cool to 4°C	7 / NA
TKN	351.2	250mL / Plastic	4oz. glass jar	H2SO4 / cool to 4°C	28 / 28
TOC	SM5310C / 9060	250mL / Plastic	4oz. glass jar	H2SO4 / cool to 4°C	28 / 28
TOC	Lloyd Kahn	NA	4oz. glass jar	none / cool to 4°C	NA / 14
TPH	1664A	1L / Glass	4oz. glass jar	H2SO4 / cool to 4°C	28 / NA
TS	SM2540B	500mL / Plastic	NA	none / cool to 4°C	7 / NA
TSS	SM2540D	500mL / Plastic	NA	none / cool to 4°C	7 / NA
Turbidity	180.1	250mL / Plastic	NA	none / cool to 4°C	48 hrs / NA
Volatile Fatty Acids	5560C	250mL / Plastic	NA	none / cool to 4°C	14 / NA
Metals Methods					
Mercury, Total	7470A, 7471A / B	250mL / Plastic	4oz. glass jar	HNO3 / cool to 4°C ⁴	28 / 28
Hexavalent Chromium	7196A	500mL / Plastic	4oz. glass jar	none / cool to 4°C	24 hrs / 28
Metals, Total / Dissolved	200.7 / 6010B / C	250mL / Plastic	4oz. glass jar	HNO3 / cool to 4°C ⁴	180 / 180
Organic Methods					
Glycols or Methanol	8015B / C	3-40mL vials	4oz. glass jar	none / cool to 4°C	14 / 14
Chlorinated Acids / Herbicides	8151A	1L / Glass	4oz. glass jar	none / cool to 4°C	7 / 14
EDB / DBCP	8011 / 504.1	3-40mL vials	NA	(HCl optional) / cool to 4°C	14 / NA
Pesticides	8081A / B	1L / Glass	4oz. glass jar	none / cool to 4°C	7 / 14
PCBs	8082 / 8082A	1L / Glass	4oz. glass jar	none / cool to 4°C	7 / 14
Volatiles	8260B	3-40mL vials	Encore / Terracore / 4oz jar	HCl (W)/ 2 Water or 2 NaHSO4 and 1 MeOH / cool to 4°C	14 / 14 ³
Explosives	8330 / 8330A / 8330B (discrete)	1L / Glass	4oz. glass jar	none / cool to 4°C	7 / 14
Explosives	8330B_KG (MIS)	1L / Glass	1 gallon plastic bag	none / cool to 4°C	7 / 14
Perchlorates	6850	250mL / Plastic	4oz. glass jar	none / cool to 4°C	28 / 28
3 – Soil En Core® and unpreserved soil 40mL VOA vial aliquots (5 grams soil to 5 milliliters water) require sodium bisulfate preservation or freezing within 48hours of sampling.					
4 - Metals waters received unpreserved and subsequently preserved in-house must be held 24 hours prior to preparation.					

<u>Attachment 2 – METHOD PRESERVATION SUMMARY</u>		<u>CONTAINERS</u>		<u>PRESERVATION</u>	<u>HT (days)</u>
<u>ANALYSIS</u>	<u>METHOD</u>	<u>WATER</u>	<u>SOIL</u>	~soils get NO preservation~ ¹	<u>Water / Soil</u>
<u>Organic Methods (contd.)</u>					
Semi-volatiles	8270C / D	1L / Glass	4oz. glass jar	none / cool to 4°C	7 / 14
MEE	RSK-175	3-40mL vials	NA	HCl / cool to 4°C	14 / NA
GRO	8015B / C	2-40mL vials	MeOH / 4oz jar	H2SO4 / cool to 4°C (HCl alternate)	14 / 14
DRO	8015B / C	1L / Glass	4oz. glass jar	H2SO4 / cool to 4°C (HCl alternate)	7 / 14
TNEPH	8015B / C	1L / Glass	4oz. glass jar	H2SO4 / cool to 4°C (HCl alternate)	7 / 14
FLPRO	FLPRO	1L / Glass	4oz. glass jar	H2SO4 / cool to 4°C (HCl alternate)	7 / 14
TCLP Volatiles	1311_8260	3-40mL vials	4oz. glass jar	HCl / cool to 4°C	14 / 14
TCLP Semi-volatiles	1311_8270	1L / Glass	4oz. glass jar	none / cool to 4°C	14 / 14
TCLP Metals	1311_6010 / 7470 / 7471	250mL / Plastic	4oz. glass jar	none / cool to 4°C	14 / 14
TCLP Pesticides	1311_8081	1L / Glass	4oz. glass jar	none / cool to 4°C	14 / 14
TCLP Herbicides	1311_8151	1L / Glass	4oz. glass jar	none / cool to 4°C	14 / 14
1 – Volatile soils can be field sampled / preserved in 40mL VOA vials with 5g to 5ml sodium bisulfate solution or methanol to provide a 14 day holding time when cooled to 4°C.					
2 - Field tests to be performed within 15-minutes, usually samples are OOH when received, upon receipt test as soon as possible.					
3 – Soil En Core® and unpreserved soil 40mL VOA vial aliquots (5 grams soil to 5 milliliters water) require sodium bisulfate preservation or freezing within 48hours of sampling.					
4 - Metals waters received unpreserved and subsequently preserved in-house must be held 24 hours prior to preparation.					

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

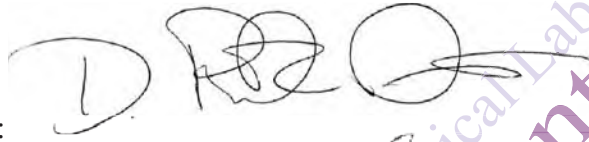
QUALITY SYSTEMS: QS14 REVISION #: 08 EFFECTIVE DATE: 20130123

Reviewed by Rick Davis 03/10/14 - found to need no change.

ANALYTICAL LABORATORY WASTE DISPOSAL

APPROVALS:

Lab Director:




Date: 01/23/2013

Data Quality Manager:



Date: 01/23/2013

Sample Receiving Manager:



Date: 01/23/2013

Revision R08 20130123 – summary of changes:

- Additional information added to section IV.B. concerning handling of soil samples for disposal.
- Labeled laboratory floorplan added to the end of the SOP as Attachment A and referenced in section IV.B.

Changes to this Revision – R07 20120430

- Revision to QS14 R06 dated 08/31/2010.
- Updates have been made to all sections of this SOP. Training covering all aspects is required.

Changes to this Revision – R06 08/31/2010

- Revision to SOP405 R05 dated 6/23/2009.
- Changed the document control and named this as QS14 R06.
- Minor cosmetic/grammatical changes made.

Analytical Laboratory Waste Disposal Standard Operating Procedure

I. SCOPE AND APPLICATION:

Laboratory waste includes excess client sample waste and waste that is generated while performing an array of analytical services, some of which are hazardous. These wastes must be disposed of in a manner that is safe, cost efficient and in accordance with hazardous waste regulations.

A. Wastes can be broken down into the following categories:

1. Unused portions of actual samples received from outside clients
 - a. Unused aliquots of completed water samples
 - b. Unused aliquots of completed non-aqueous samples
2. Soils from quarantined areas
3. All other soils, sediments, building debris, wipes, oils, etc.
4. Hazardous waste generated within the laboratory as part of numerous analytical procedures.

II. SUMMARY OF PROCEDURES:

A. There are four options for disposing of unused sample portions:

1. Return completed samples and any generated waste from these samples to the client
2. Disposal of samples after confirming that they are non-hazardous.
3. Disposal through a waste vendor in either a sealed drum or lab pack.
4. Treat the sample to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

B. There are two options for disposing of laboratory generated waste:

1. Disposal through a waste vendor in either a sealed drum or lab pack. The waste must be stored properly until transported off site.
2. Treat the waste to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

III. EQUIPMENT/APPARATUS:

- A. Proper safety equipment should be in good working condition. This includes gloves, lab coats, safety glasses/goggles, and face shields.
- B. USDOT approved drums for storing and shipping hazardous waste.
- C. Fume hoods.
- D. 5 gallon waste containers

IV. PROCEDURE

Waste disposal is done under the management and coordination of the Sample Receiving Manager, Section Supervisors, Safety Coordinator and Lab Director.

A. Disposal of completed aqueous samples:

Completed samples are kept in cold storage for a minimum of 45 days from receipt and sample extracts are held for 90 days minimum from receipt. Engineering support projects involving CLP work, litigation cases, etc. may be saved for longer than 45 days at the request of the project manager.

1. The majority of the water samples (ground, surface and drinking) are non-hazardous and are disposed of by pouring them down the sink in the hooded area in sample receiving as follows:
 - a. Make sure that the sash is closed far enough to produce sufficient ventilation.
 - b. Turn on the tap water to supply copious rinse for sample disposal.
 - c. Wear proper safety equipment including safety glasses (face shield, if necessary), lab coat, and gloves.
 - d. **Be alert to potential problems: for example, separate cyanide waste from acid waste. Neutralize acid waste that will be poured down the drain and don't mix waste/samples. Also, look for things such as phase separation, odd color, odor, etc. Check with the Section Supervisor or Safety Coordinator before disposing of any questionable samples.**
 - e. Leave tap water running when samples are poured out and for approximately 10 minutes following disposal in order for sufficient flushing and dilution to take place.
 - e. Rinse all containers and discard into the trash.
 - f. **Document disposal in the bound aqueous sample disposal logbook with date, initials, workorder and any appropriate comments.**
2. If water samples are hazardous, one of the following steps must be taken:
 - a. Samples may be returned to the client. If you plan to ship the unused portion back to the client, check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations. **If the samples are not returned to the client they must be stored properly until picked up by a waste vendor.**
 - b. Treat the sample to make it non-hazardous. One example of this is if the sample is highly corrosive, the pH may be adjusted.
 - c. Store the sample properly until either a sealed drum or lab pack is sent out.
 - d. **Document disposal in the bound aqueous sample disposal logbook with date, initials, workorder and any appropriate comments concerning treatment, lab-pack, etc.**

B. Disposal of completed non-aqueous samples:

Note – for purposes of the USDA permit, see attachment A for a floorplan of the laboratory located at 621 Mainstream Drive, Suite 270, Nashville, TN, 37228 (GPS coordinates N36° 11.9094' and W086° 48.9253'). Emergency contacts are located on the laboratory phone list which is to be posted on the outside of the hood for the oven used to treat soils.

The majority of non-aqueous samples are soils and sediments. Although there may also be building debris, wipes, oils, and occasionally product type samples.

1. Soil samples taken at a depth of three feet or less from areas, which have been quarantined by the US Department of Agriculture (USDA), must first be treated at the laboratory to prevent the spread of any plant pests. For simplicity, all soils are treated as quarantined soils. The USDA has detailed treatment procedures from which we use the following:
 - a. The sample is heated to 180°C (356°F) in a vented oven for a minimum of two hours. **USDA requirement is 121°C-154°C (250°F-309°F) for 2 hours or 154.4°C-192.6°C (310°F-379°F) for 30 minutes. We choose higher temperature with longer bake times for certainty.**
 - b. After heating the samples they are removed from the oven to cool and then placed in a sealed drum and destroyed.
 - c. **All samples disposed of in this manner must be documented in the bound soil treatment/disposal logbook with date, initials, workorder(s), oven ID, temperature, quantity (oz) and any appropriate comments.**
2. If non-aqueous samples are hazardous, one of the following steps must be taken.
 - a. Samples may be returned to the client. If you plan to ship the unused portion back to the client, check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations.
 - b. **If the samples are not returned to the client they must be stored and labeled properly until picked up by a waste vendor.**

C. Disposal of laboratory generated waste:

Generated waste is stored until a waste pick up occurs. These areas must be maintained properly by using satellite stations within several laboratory areas. These stations must be labeled appropriately and properly maintained.

****Note: Laboratory generated solvent waste is transferred to the appropriate Satellite Solvent Waste Drum weekly or as deemed necessary. Disposal of solvent waste is done under the direction of the Laboratory Director or Safety Coordinator. The solvent waste drums are located outside the back of the building inside the caged fence and only authorized laboratory staff add waste solvent to these drums.**

1. Waste handling and disposal within each laboratory section:

NOTE: Each laboratory analyst and section supervisor is responsible in assuring that **handling** operations (within their area) are being followed according to the laboratory requirements.

a. General Chemistry/Inorganic

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If any questions remain unanswered regarding waste disposal within your specific area contact the Section Supervisor or the Safety Coordinator.

- Acid waste is neutralized by using sodium hydroxide. Once the pH of the acid waste has been neutralized (pH 5-9), the acid waste is then poured down a sink within hooded ventilation followed by 10 minutes of flushing with tap water. The amount of acid waste treated, the amount of sodium hydroxide used to neutralize the acid waste, final pH, date performed and the date disposed of is then recorded in an acid waste neutralization log book along with the initials of the person performing the neutralization/disposal.
- Hach Ferrous Iron vials are placed into the acid waste area for disposal. The contents of these vials are not hazardous but they are in sealed glass vials, so being very careful with respect to cuts is extremely important and the Safety Coordinator or his designee will handle this task.
- Chemical Oxygen Demand (COD) vials have to be dealt with as a special waste due to its contents. Since we use so very few of these, we will store all of these until we have adequate volume to be handled by a designated hazardous waste vendor. It will take an estimated 3-4 years to complete this exercise.

b. Metals

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If any questions remain unanswered regarding waste disposal within your specific area contact the Section Supervisor or the Safety Coordinator.

- **Concentrated acid waste, aqueous sample waste digestates, and old unused calibration standards (>2% by volume)** are taken to the Acid Satellite Station located on the shelving to the right of the back Wet Chem door. The shelving has signs to identify what waste goes in each area.
- **Non-aqueous sample digestate wastes** are decanted off the soil/solid samples into the Acid Satellite Station located on the shelving to the right of the back Wet Chem door.

The shelving has signs to identify what waste goes in each area.

- **Cr6 digestates as with all concentrated metal/acid waste are also placed in the Acid Satellite Waste Station** located on the shelving to the right of the back Wet Chem door. The shelving has signs to identify what waste goes in each area.

c. Organic Extraction Laboratory Area

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If any questions remain unanswered regarding waste disposal within your specific area contact the Section Supervisor or the Safety Coordinator.

- **Concentrated acid waste** is taken to the Acid Satellite Waste Station located on the shelving to the right of the back Wet Chem door. The shelving is labeled to show what waste is stored in each area.
- **Non-chlorinated solvent waste** (Acetone, Ether, Hexane, and Methanoletc...) is poured into the Non-Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.
- **Chlorinated solvent waste** (Methylene Chloride, Chloroform, chlorinated standard and spike waste) is poured into the Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.
- **Aqueous sample waste from extracted samples (after the extraction solvent has been removed) is poured down the drain and flushed with copious amount of tap water.**
- **Sodium sulfate** waste is dumped into a waste container under an extraction laboratory hood and left overnight or until evaporated. Then the waste is discarded into the trash.
- **Unused stock and working standards** are emptied into the chlorinated or non-chlorinated solvent waste bottle. The empty vials are discarded in the glassware waste container.

d. Gas Chromatography (GC)/High Performance Liquid Chromatography (HPLC) Laboratory:

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If any questions remain unanswered regarding waste disposal within your specific area contact the Section Supervisor or the Safety Coordinator.

- Unused stock standard, unused working standards, sample extract autosampler vials and sample extract vials are separated by the solvent and discarded into the designated 5 gallon waste container located in the GC/HPLC Laboratory.
- **Acid cleaned extracts** are separated and provided to the sample receiving manager. They are later combined into a separatory funnel and the acid layer separated from the solvent. The acid portion is placed in the Acid Satellite Waste area located between Wet Chem and Extractions while the solvent waste is discarded into the appropriate solvent waste bottle (chlorinated/non-chlorinated waste) located in the hood in the organic extraction laboratory.

e. Gas Chromatography/Mass Spectrometry

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If any questions remain unanswered regarding waste disposal within your specific area contact the Section Supervisor or the Safety Coordinator.

- **Volatile sample, standard, and reagent waste:**
Instrument Waste - Aqueous sample waste is collected in waste bottles via waste lines from the instruments. The bottles are emptied into waste containers and poured down the drain with flushing with tap water for an additional 10 minutes following disposal. A small amount of methanol used to clean glassware is also dumped into the waste container and poured down the drain.
Unused stock and working standards are separated by solvent and discarded appropriate designated 5 gallon waste container located in the GC/MS or GC/HPLC Laboratory for later consolidation into lab packs..
- **Semi-volatile sample and standard waste disposal:**
Unused stock standard, unused working standards, autosampler vials and extraction vials are discarded into the appropriate designated 5 gallon waste container located in the GC/MS Laboratory for later consolidation into lab packs.

D. Consolidation of satellite waste for contractor disposal:

In conjunction with the Safety Coordinator, the Sample Receiving Manager and the Lab Director are responsible to coordinate waste disposal operations with outside waste disposal contractors.

1. Solvent waste from the areas discussed above is periodically consolidated into a drum located outside the back of the building inside the caged fence. Drums designated either chlorinated or non-chlorinated solvent waste are available to receive the appropriate solvent waste. When the drums become full (fluid surface six inches below the top of the drum), an

authorized hazardous waste contractor must be scheduled to remove them for proper waste disposal.

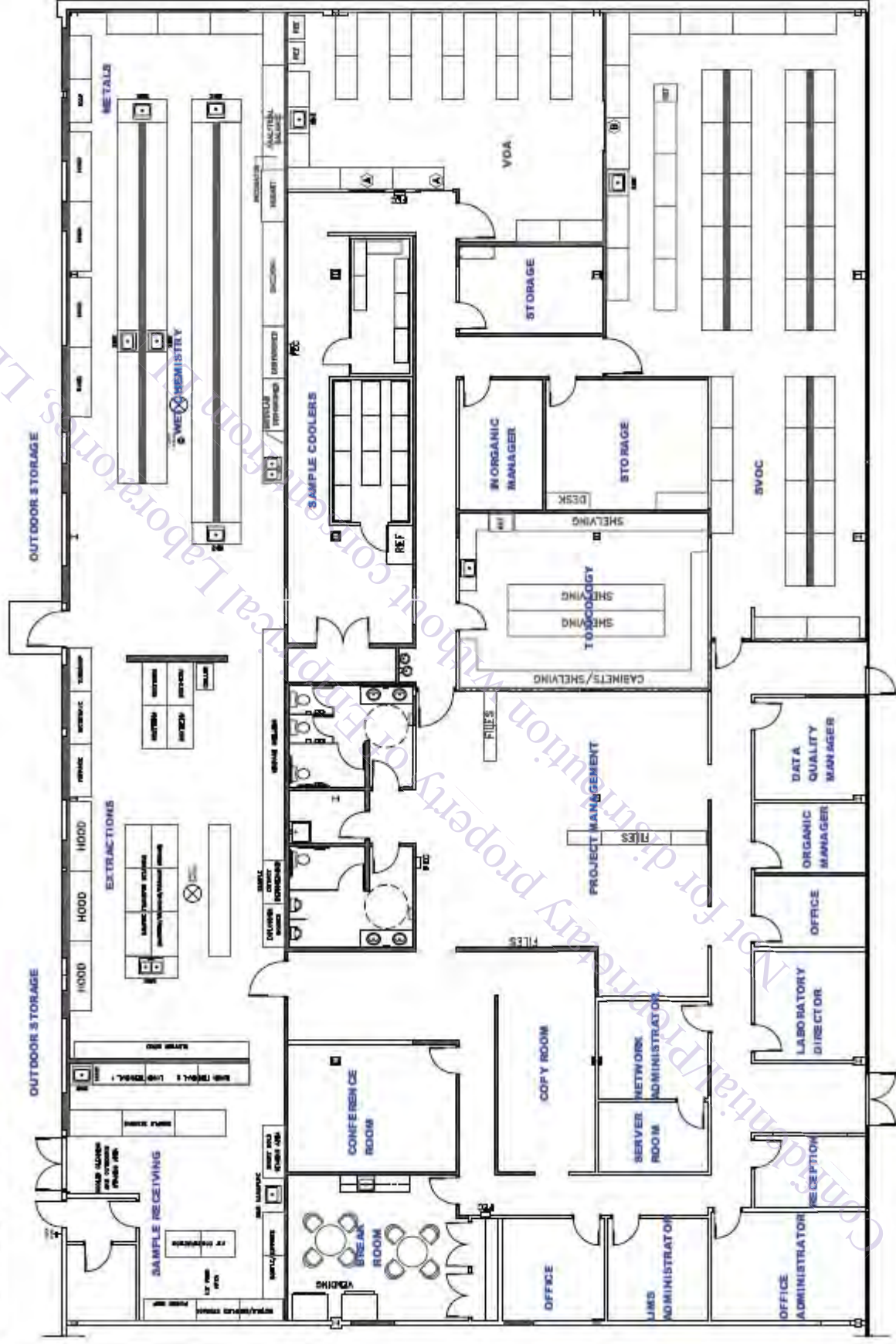
2. The Acid Waste drum is also disposed through the authorized hazardous waste contractor once the drum is full to the level of six inches below the top of the drum.
3. Consolidated autosampler and standard vials are periodically Lab-Packed in drums and disposed of through an authorized hazardous waste contractor.
4. The Safety Coordinator and the Sample Receiving Manager, in conjunction with the Lab Director, will administer the Waste Disposal Program and maintain current information to track quantities of waste generated and stored on-site.

It is the continuous objective of our laboratory to find ways to decrease the amount of waste generated.

Attachment A: See next page for laboratory floor plan.

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EMPIRICAL LABORATORIES, LLC
621 MAINSTREAM DRIVE, SUITE 270
NASHVILLE, TN 37228



MAIN ENTRANCE

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

INORGANICS: SOP 417

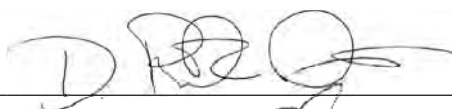
REVISION #: 06

EFFECTIVE DATE: 20130411

**TRACEABILITY AND EXPIRATION DATES OF TEST -RELATED CHEMICALS FOR
ORGANIC AND INORGANIC METHODS**

APPROVALS:

Lab Director:



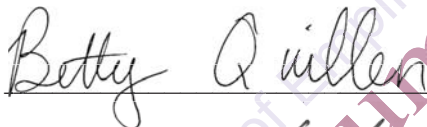
Date: 04/11/2013

Data Quality Manager:




Date: 04/11/2013

Section Supervisor:



Date: 04/11/2013

Section Supervisor:



Date: 04/11/2013

Changes Summary

Revision 06, 04/11/2013

- Updated label requirements for reagents/standards.
- Added LIMS documentation requirements including record of reagents for bench sheets.
- Added COA documentation and scanning for review within LIMS.

Revision 05, 10/31/11

- The SOP is an update from Revision 04 revised 05/16/11.
- Clarification added to section 3 for manufacturer expiration dates listed as MM/YY only – last day of the month assigned.
- Corrective action designated in section 3 for solutions incorrectly made – discard solution, inactive LIMS ID and include in LIMS standard comments “Made incorrectly – not to be used!”.
- QA, Safety and Waste Management SOP references added to section 3.

Revision 04, 05/16/11

- The SOP is an update from Revision 03 last reviewed 09/0/10
- Updates to reference the new LIMS and standard entry are included
- Updates to reference order receipt confirmation procedure

TRACEABILITY AND EXPIRATION DATES OF TEST -RELATED CHEMICALS FOR ORGANIC AND INORGANIC METHODS

1. Scope and Application

- 1.1. This Standard Operating Procedure applies to all reagents and standard solutions that are routinely handled in the laboratory.

2. Summary of Approach

- 2.1. This SOP distinguishes different types of reagents, standards, and chemicals and describes the approach to identification and labeling of the containers. Proper labeling must include identification of the expiration date of the item in question. Specific criteria are given to serve as guidelines in the decision-making process and assignment of an enforceable expiration date.
- 2.2. The laboratory's LIMS system allows for complete documentation and traceability of reagents and standards used within the laboratory. All certificates received with standards must be marked with the LIMS ID and forwarded to the administration department for scanning/saving in order to be available for review within LIMS. All reagents received or prepared are recorded within the LIMS and added to the batch sheets to ensure traceability to the associated samples.

3. QA/QC Issues/Safety/Waste Management

- 3.1. The application of criteria under Section 3 is intended as a set of guidelines to standardize the holding times of standard solution and test reagents. The following QA/QC considerations must be also evaluated when making a decision that affects the assignment or enforcement of expiration dates.
 - 3.1.1. The day-to-day handling of solutions, standards, reagents or chemicals may reveal some kind of deterioration (e. g., color, smell, precipitation). Inform Section Supervisor and decide on appropriate corrective action.
 - 3.1.2. Any blatant mishandling of a solution by the previous user (e. g., container kept without preservation and at room temperature for an extended period of time) must trigger corrective action and replacement of the item.
 - 3.1.3. Specific projects, clients or methods may call for the preparation of a "fresh" reagent or a "customized" standard solution. Consult the corresponding SOP and verify interpretation with the Section Supervisor.
 - 3.1.4. Any evidence of lack of traceability when using a solution for the first time may require replacement or regeneration of the item. Consult with Section Supervisor.
 - 3.1.5. Where the manufacturer expiration date reflects only MM/YY, the laboratory will assign the expiration date as the last day of the designated month.
 - 3.1.6. Where a solution is incorrectly made (example – inappropriate solvent used), the solution will be removed from use, the LIMS ID will be inactivated and the LIMS comments will reflect “Made incorrectly – not to be used!”. This is to prevent accidental use of the “recipe” for making up a subsequent standard.
 - 3.1.7. All standards/reagents will have an associated LIMS ID and be added to batch sheets to ensure traceability to the associated samples.
 - 3.1.8. All certificates of analysis will be marked with received date and LIMS ID then forwarded to the administration department for scanning/saving in order to be available for review within LIMS. Each container of a standard will be assigned an individual LIMS ID.

- 3.2 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.
- 3.3 Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed labwide.
- 3.4 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

4. Procedure

- 4.1. The actual purchasing of laboratory supplies and initial vendor approval will proceed as detailed in SOP 400. Also detailed in SOP 400 is the green sheet (purchase order) confirmation of items received versus what was ordered. This procedure must be followed with each laboratory supply purchase.
- 4.2. **Reagent container received, when reagent not used to prepare primary nor secondary standards** (order \leq 1yr supply if possible).

4.2.1. Dry/Solid

- 4.2.1.1. Record on container
 - 4.2.1.1.1. Date received (R: MM/YY)
 - 4.2.1.1.2. Date opened (O: MM/DD/YY, to be filled in)
 - 4.2.1.1.3. Date expired (E: MM/YY, 5 yrs unless stated otherwise by manufacturer)
 - 4.2.1.1.4. Assigned LIMS ID

4.2.2. Liquid/Solution/Suspension/Multiphase

- 4.2.2.1. Record on container
 - 4.2.2.1.1. Assigned LIMS ID.
 - 4.2.2.1.2. Date received (R: MM/YY)
 - 4.2.2.1.3. Date opened (O: MM/DD/YY, to be filled in)
 - 4.2.2.1.4. Date expired (E: MM/YY, 3 yrs unless stated otherwise by manufacturer)
 - 4.2.2.2. Check purity of nitric and hydrochloric acids for ICP metals
 - 4.2.2.2.1. Prepare aliquot at concentration for analysis and send through login procedure
 - 4.2.2.2.2. Report analysis as a sample
 - 4.2.2.3. Check methylene chloride, hexane and ether purity, as required by client.
- 4.3. **Reagent container received, when reagent used to prepare primary or secondary standards** (order \leq 1yr supply if possible).

4.3.1. Dry/Solid

- 4.3.1.1. Record on container
 - 4.3.1.1.1. Date received (R: MM/YY)
 - 4.3.1.1.2. Date opened (O: MM/DD/YY, to be filled in)
 - 4.3.1.1.3. Date expired (E: MM/YY, 3 yrs unless stated otherwise by manufacturer)
 - 4.3.1.1.4. Assigned LIMS ID

4.3.2. Liquid/Solution/Suspension/Multiphase

- 4.3.2.1. Record on container
 - 4.3.2.1.1. Date received (R: MM/YY)
 - 4.3.2.1.2. Date opened (O: MM/DD/YY, to be filled in)
 - 4.3.2.1.3. Date expired (E: MM/YY, 2 yrs unless stated otherwise by manufacturer)
 - 4.3.2.1.4. Assigned LIMS ID
- 4.3.2.2. If supplied, submit the manufacturer's certificate with LIMS ID for filing.

4.4. Stock standards for calibration curves or laboratory control samples (LCS)

4.4.1. Record in Element

- 4.4.1.1. LIMS ID of source.
- 4.4.1.2. True value of analyte
- 4.4.1.3. Details of preparation
- 4.4.1.4. Date prepared (Prep: MM/DD/YY)
- 4.4.1.5. Date expired (E: MM/DD/YY; unless indicated differently by SOP, it will be 6 months or 10 times the EPA sample holding time, whichever shorter)
- 4.4.1.6. Preparation analyst
- 4.4.1.7. Preservation
- 4.4.1.8. Storage requirements (container, temp., etc.)

4.4.2. Record on container

- 4.4.2.1. Assigned LIMS ID.
- 4.4.2.2. Concentration
- 4.4.2.3. Date prepared (Prep: MM/DD/YY)
- 4.4.2.4. Date expired (E: MM/DD/YY; unless indicated differently by SOP, it will be 6 months or 10 times the EPA sample holding time, whichever shorter)

4.5. Calibration standards generated from dilution of stock standard.

4.5.1. Record in Element

- 4.5.1.1. True value of analyte
- 4.5.1.2. Details of preparation including source standard.
- 4.5.1.3. Date prepared (Prep: MM/DD/YY)
- 4.5.1.4. Date expired (E: MM/DD/YY; unless indicated differently by SOP, it will be 3 months or 5 times the EPA sample holding time, whichever shorter)
- 4.5.1.5. Preparation analyst
- 4.5.1.6. Preservation
- 4.5.1.7. Storage requirements (container, temp., etc.)

4.5.2. Record on storage container

- 4.5.2.1. Assigned LIMS ID.
- 4.5.2.2. Concentration
- 4.5.2.3. Date prepared (Prep: MM/DD/YY)
- 4.5.2.4. Date expired (E: MM/DD/YY; unless indicated differently by SOP, it will be 3 months or 5 times the EPA sample holding time, whichever shorter)

Note: If a calibration standard is also to be used as a check standard for a curve used longer than 1 day assign an Element # as in 4.3.1 and 4.3.2 above, but maintain expiration criteria under 4.4.1(e).

4.6. Reagent, prepared but not used for calibration standards or LCS

4.6.1. Record in Element

- 4.6.1.1. Test for which the reagent will be used (if you use a prepared reagent for a test not listed in Element, record at the time of first use)
- 4.6.1.2. Manufacturer of reagent
- 4.6.1.3. Lot# of source reagent
- 4.6.1.4. Expiration date of source reagent
- 4.6.1.5. Element# of reagent solution
- 4.6.1.6. Concentration of reagent solution, where appropriate

- 4.6.1.7. Details of preparation
- 4.6.1.8. Date prepared (Prep: MM/DD/YY)
- 4.6.1.9. Date expired (E: MM/DD/YY, 6 months unless indicated differently in SOP of related test)
- 4.6.1.10. Preparation analyst
- 4.6.1.11. Preservation
- 4.6.1.12. Storage requirements (container, temp., etc.)

4.6.2. Record on container


- 4.6.2.1. Assigned LIMS ID.
- 4.6.2.2. Concentration of reagent solution, where appropriate
- 4.6.2.3. Test for which reagent used (if you use a prepared reagent for a test not listed in the book, record at the time of first use)
- 4.6.2.4. Date prepared (Prep: MM/DD/YY)
- 4.6.2.5. Date expired (E: MM/DD/YY, 6 months unless indicated differently in SOP of related test)


**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

ADMINISTRATIVE: SOP 400 REVISION #: 08 EFFECTIVE DATE: 20130805

**LABORATORY ADMINISTRATIVE ASSISTANT
SOP FOR PURCHASING**

APPROVALS:

Lab Director:  Date: 08/05/2013

Data Quality Manager:  Date: 08/05/2013

Section Supervisor:  Date: 08/05/2013

Changes Summary

Revision 08, 08/05/13

- Revised Purchase Order Form and moved vendor list to next page.

Revision 07, 10/18/12

- Added Section 4.3 on bid procurement.
- Added Figure A.

Revision 06, 08/20/12

- Updated location of Vendor Account Information within the network.
- Updated SOP to reflect the use of a Master Parts List.
- Updated Preferred Vendor list to reflect recent purchasing history.

Revision 05, 05/16/11

- The SOP is an update from Revision 04 dated 06/01/08.
- Updates to reference laboratory vendor approval.

LABORATORY ADMINISTRATIVE ASSISTANT (AA) SOP FOR PURCHASING

1. Purchase Orders/Requisitions are completed to keep a detailed record of all purchases, track expenses and to ensure prompt payment to valid vendors.
2. Completed Purchase Requisition forms (Figure A) will be placed in the designated tray in the Front Office and must include the department's manager approval.
3. Find the vendor's phone number along with their account number located on the Vendor List tab in V:\Admin\Front Office. Verify pricing with the Master Parts List, located in V:\Admin\Front Office, and update prices as necessary.
4. Contact the vendor.
 - 4.1. In the vendor list referenced in item 3 above, find the vendor's phone number or website address.
 - 4.2. Request and document both *list* and *our* price and populate the Purchase Order form to include subtotals.
 - 4.3. Competitive bid procurement will be at the discretion of the Laboratory Director/designee for laboratory supplies/equipment and Office Manager/designee for all other supplies.
5. Once prices are obtained, the order must receive executive approval.
 - 5.1. For laboratory supply purchases, approval is required by the Laboratory Director.
 - 5.1.1. Vendors used in this area are selected based on initial conformance to specifications within each analytical method or regulatory program requirement. These specifications take the form of ACS certified chemicals and certified standards/reagents with COAs contained within. PT's and balance/thermometer vendors are another critical item that require ISO17025 certification from the vendor itself. Also, many of our everyday use supplies are considered general consumable supplies which do not require any conformance standards.
 - 5.1.2. Our vendors are selected on several factors:
 - 5.1.2.1. Specific method and/or regulatory program (i.e. NELAC, DoD QSM) requirements
 - 5.1.2.2. Service/responsiveness
 - 5.1.2.3. Amount of stock on hand
 - 5.1.2.4. Price and timely delivery of purchased item
 - 5.2. For office, janitorial, and IT supply purchases, approval is required by Office Administrator.
 - 5.2.1. Vendors used in these areas are selected based on the best value (price and timely delivery) to the Company.
6. Once executive approval is obtained, a Purchase Order number is assigned by obtaining the number from the PO Book referenced in item 3 above.
7. Place the order.
 - 7.1. Call vendor or place the order online.
 - 7.1.1. Vendor may ask you to verify company information such as Company Name, Telephone Number, Your Name, Purchase Order #, etc.
 - 7.1.2. You will be asked for part/item numbers and quantity.
 - 7.1.3. Pricing will be given again so verify that the first price you obtained is accurate.
 - 7.1.4. If this is a "rush" order, needing items next day or 2nd day, always use our Fed Ex account.
 - 7.1.5. Verify when the order will be received. If any items are on back order, discontinued, or out of stock, call the employee requesting the order and see if this is acceptable to them. They might want to order the item from another vendor. Regardless of the outcome, always write these comments down.
 - 7.2. Once the order is placed, scan the Purchase Order form to V:\Admin\Front Office\Purchase Orders\PO Scans, and add a hyperlink of the form to the PO book referenced in item 3 above.
 - 7.3. The Purchase Order form is then given to and kept by the employee receiving the order until the order arrives. The employee will then verify that all items were received as ordered and all product/material specifications to include CCAs are met by signing the acknowledgment section at the bottom of the Purchase Order form and by initialing each packing/shipping slip and attaching them to the form, returning all documents to the Front Desk.
8. Final Purchase Order forms as outlined in section 7.3 will be submitted to the Corporate office every week.

[illegible]

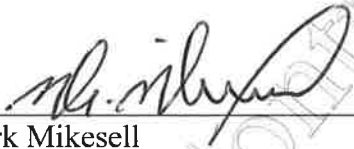
LABORATORY SUPPLIES PREFERRED VENDOR LIST

Absolute Standards, Inc
ABS
AccuStandard, Inc.
Agiant Packaging
Agilent Technologies
A&L Compressed Gases
Associated Design
Biotage
Chem Service Inc
Dionex
DiscountVials
Environmental Express
ERA (Environmental Resource Associates)
ESS
Fisher Scientific Company
HACH
High- Purity Standards
Metrohm
Nashville Barrell & Drum
NSI Environmental Solutions, Inc
Perkin Elmer
Phenomenex
QEC (Quality Environmental Containers)
Reeves Glass
Restek Corporation
SCP Science
Supelco (Sigma-Aldrich family of companies)
ULine
Ultra Scientific, Inc
Vici Metronics
VWR Scientific
WebStaurant

Pace Analytical Energy Services, LLC.

Analytical Method AM20GAx Standard Operating Procedure for the Analysis of Biodegradation Indicator Gases

Controlled Copy No. _____



Mark Mikesell
Laboratory Manager

Signature of Final Approval:



Patrick McLoughlin, Ph.D.
Technical Director



9-9-13
Effective Date

1.0 Scope and Application

Method AM20GAx is used to determine the concentration of biodegradation indicator gases in vapor samples. Specifically, Method AM20GAx is used to determine the dissolved concentration of the following gases:

Gases	CAS Number
Acetylene	74-86-2
Carbon dioxide	124-38-9
Oxygen	7782-44-7
Nitrogen	7727-37-9
Hydrogen	1333-74-0
Methane	74-82-8
Ethane	74-84-0
Ethene	74-85-1
Propane	74-98-6
Propene	115-07-1
n-Butane	106-97-8
i-Butane	75-28-5
Carbon Monoxide	630-08-0
Total Inorganic Carbon*	

*Total inorganic carbon (TIC) is converted to carbon dioxide using the steps outlined in SOP-PM01. The sample is then analyzed for carbon dioxide according to this SOP. Any differences in method are specified in the appropriate section.

This method is recommended for use by, or under the supervision of, analysts experienced in sample preparation, the operation of gas chromatographs and in the interpretation of chromatograms.

2.0 Method Summary

The sample gas is analyzed with a gas chromatograph capable of simultaneous analysis of all of the target analytes from a single gas sample. A single injection of gas from integral, simultaneously filled sample loops is used to assure consistent injection volume. The permanent gases are analyzed using a thermal conductivity detector (TCD). The light hydrocarbons are analyzed using a flame ionization detector (FID). Hydrogen is analyzed using a reduction gas detector (RGD). The data are transferred to a microcomputer, converted to digital format, stored, and processed using a chromatography data system.

2.1 Definitions

Batch: A batch consists of twenty or fewer client samples, submitted to the laboratory for analysis.

Instrument Flush: The front end of the sample loop is flushed with ultra high purity helium injected into the loop directly from the cylinder to remove possible interference by ambient air and to avoid cross contamination between samples.

Method Blank: An injection analyzed by all three detectors that consists of ultra high purity helium. The method blank is free from the analytes of interest

Laboratory Control Sample: A sample of laboratory grade deionized water spiked with verified known amounts of analytes. A LCS is used to assess the performance of the measurement system.

Matrix Spike and Matrix Spike Duplicate: A sample prepared by adding a known concentration of target analyte to a specific amount of sample. Matrix spikes are used to determine the effect of sample matrix on a method's recovery efficiency.

3.0 Apparatus and Materials and Operating Conditions

3.1 Apparatus

Gas Chromatograph: The chromatographs designed and built by Microseeps are equipped with multiple packed columns and multi-port valves, a TCD, a FID, a RGD, and multiple sample loops. The FIDs, which were also built by Microseeps, are of a special design that allows considerably more sensitivity than commercially available models. This instrument provides rapid turn-around for consecutive analyses and a clean baseline for accurate, reproducible results.

3.2 Materials

- Sample vials (Supelco, Inc, Alltech; 40ml clear and amber glass vials with butyl septa or equivalent)
- Syringes: Disposable Plastic (BD Medical; 3,5,10,30 and 60ml or equivalent)
- Headspace vials (Supelco, Inc./Alltech; 10 and 20ml clear glass with butyl septa or equivalent)

3.3.1 Interferences

The most likely source of "interference" is ambient air. Due to the relatively high concentrations of oxygen and nitrogen, a very small amount of air as a contaminant will dramatically affect the

results. The analyst must take great care to ensure that air is flushed from the syringe before sample preparation and that no air has entered the syringe or needle prior to injection of the sample into the gas chromatograph.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. An unrestricted flow (Instrument flush) of pure carrier gas from a 10 psig source should be allowed to flow through each sample loop for a minimum of 2 minutes prior to each analysis.

As required, the analyst should demonstrate the absence of carryover contamination by analysis of the contents of the sample loop when purged with carrier gas. This demonstration should be performed when carryover contamination is suspected (after high samples). In the event that 'ghost peaks' (peaks similar to previous sample) appear when a pure carrier gas sample is analyzed (method blank), measures should be taken to eliminate the carryover contamination.

4.0 Reagents

- Ultra High Purity Gases (Helium, Nitrogen, Hydrogen and compressed air)
- Certified Commercial Gas Standards
- Benzalkonium chloride (BAK) solution – Prepared by dissolving 12.08 g into 1L DI water.
- Tri-sodium phosphate (TSP) – purchased as the dodecahydrate

4.1 Standard Preparation Procedures

Calibration standards are prepared by using the procedures below:

4.1. Vial Preparation

Headspace vials used for instrument calibration standards for this method are prepared as follows:

- Crimp and cap each vial, with stopper septa.
- Evacuate each vial to vacuum. Attach vials to the vacuum manifold for 10 minutes to achieve evacuation.
- Flush each vial to atmospheric pressure with the vial balance gas appropriate for the detector being calibrated. (See Table 4.1)

Table 4.1

Detector	Vial Balance Gas
FID	Nitrogen
TCD	Helium
RGD	Nitrogen

4.1.1 Preparing Calibration Standards

The instrument is initially calibrated (ICAL) using dilutions of custom certified gas mixes.

- Prepare the correct number of vials for the detector being calibrated.
- Each of the three detectors is calibrated with a gas mix from a commercial source.
- Remove the specified amount of standard by extracting it from the standard mix gas cylinder using a syringe and injecting it into a prepared vial.
- Add the specified amount of vial balance gas to the same vial.

The dilution factor of one is achieved by directly injecting the standard gas mix from the cylinder into the GC.

4.1.2 Calibration Standard Concentrations

Calibration standards are made up in the following concentrations as specified in Tables 4.1.2 A, B, C, and D. The true values of the calibration standards vary slightly from cylinder to cylinder. The values below are very close approximations. All standards are prepared using headspace vials with stopper septum or serum bottles.

Table 4.1.2 A
Light Hydrocarbons by FID
(Methane, Ethane, Ethene, Butane, Propane, Propene and Acetylene)

Stock-1000ppmv Hydrocarbon Mix in Nitrogen from Matheson Tri-Gas, or equivalent.

Stock-1000ppmv Acetylene in Nitrogen, Matheson Tri-Gas, or equivalent.

Std Level	Conc. (PPMV)	Std	Make-up Gas
Level 9	0.040	2cc (W/S#8)	248cc UHP Nitrogen (w/serum bottle)
Level 8	0.100	1cc (W/S#8)	49cc
Level 7	0.200	2cc (W/S#8)	48cc

Std Level	Conc. (PPMV)	Std	Make-up Gas
Level 6	0.500	4cc (W/S#8)	36cc
Level 5	2.00	2cc (W/S#7)	38cc
Level 4	10.0	10cc (W/S#7)	30cc
Level 3	40.0	2cc (each stock)	46cc
Level 2	125	5cc (each stock)	30cc
Level 1	500	21cc (each stock)	Into an evacuated vial
W/S#7	40.0	8cc (each stock)	184cc (w/160cc serum bottle)
W/S#8	5.00	1cc (each stock)	198cc (w/160cc serum bottle)

Table 4.1.2 B
Hydrogen by RGD

Stock-100 PPMV Hydrogen in Nitrogen, Matheson Tri-Gas, or equivalent.

Level	Conc.	Std	Make-up Gas
Working Sol #4	2.00	1cc Stock	49cc
Level 1	50.0	21cc Stock	21cc
Level 2	20.0	10cc Stock	40cc
Level 3	10.0	5cc Stock	45cc
Level 4	5.00	2cc Stock	38cc
Level 5	2.00	1cc Stock	49cc
Level 6	0.500	10cc Working Sol #4	30cc
Level 7	0.200	4cc Working Sol #4	36cc

Table 4.1.2 C
Permanent Gases by TCD
(Oxygen, Carbon Dioxide, Nitrogen, Methane, Carbon Monoxide)

Stock-Multi-component Mix at various conc. in Nitrogen, Matheson Tri-Gas, or equivalent.

Level	Std	Make-up Gas
Working Sol #5	1cc Stock	49cc

Level	Std	Make-up Gas
1	As received from cylinder	0
2	21cc Stock	21cc
3	5.0cc Stock	45cc
4	1.0cc Stock	49cc
5	0.5cc Stock	49.5cc
6	10cc Working Sol #5	40cc

Table 4.1.2 D
Permanent Gases by TCD
(Carbon Dioxide, Methane, Ethane, Ethene)

Stock-Single component sources, 100% Stock by Matheson Tri-Gas, or equivalent.

Std Level	Conc. (PPMV)	Std	Make-up Gas
Working Sol #6	20,000	5cc each comp	230cc (w/serum bottle)
Level 1	200,000 CO ₂ 100,000 MEE	10cc CO ₂ 5cc MEE	25cc
Level 2	50,000	2.5cc each comp	40cc
Level 3	10,000	25cc Working Sol #6	25cc
Level 4	2,000	5.0cc Working Sol #6	45cc
Level 5	400	1.0cc Working Sol #6	49cc

4.2 Quality Control Sample Preparation

Quality control samples are prepared as indicated below.

4.2.1 Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

The ICV and CCV are prepared from a source different from the source used to prepare the ICAL standards. The concentration of the ICV and CCV is in the middle of the calibration range and is close to that of the ICAL midpoint, but because of the nature of gas standard it is not at exactly that concentration.

4.2.2 Laboratory Control Sample (LCS) and LCS Duplicate (LCSD)

The LCS and LCSD are prepared at a mid-range concentration. The type of LCS/LCSD depends upon the original matrix of the sample. For samples that arrive as vapors, the LCS/LCSD is injected as a gas. For samples that arrive as waters, DI water is spiked with a gas mixture of target analytes and prepared the same as the samples. Table 4.2.2 below gives the true values of the LCS/LCSDs.

4.2.2.1 Total Inorganic Carbon LCS

Mix approximately 0.20g NaHCO₃ into 200ml laboratory grade DI water, prepare according to the TIC procedures outlined in PM01 and analyze in duplicate as a sample. The true value of the spike is calculated as follows:

$$\text{mg/L CaCO}_3 = \frac{\text{Mass(g)NaHCO}_3}{\text{H}_2\text{O(L)}} \times \frac{100.09}{84.01} \times (1,000,000)$$

4.2.3 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

- For water samples, MS and MSDs are prepared, analyzed, and reported when clients' request and send sufficient numbers of aliquots to prepare them (e.g. one 40 ml vial each for the MS and another for the MSD).

Table 4.2.2

Compound	Vapor LCS/LCSD (ppmv)	Water LCS/LCSD & MS/MSD	
		A	B
Methane	300.0	748	40
Ethane	100.0	38	76
Ethene	100.0	35	70
Propane	100.0	56	111
Propene	100.0	53	106
iso-Butane	100.0	73	146
n-Butane	100.0	73	146
Acetylene	-----	33	33
Carbon dioxide	50,000	117	110
Oxygen	20,000	11	9.5
Nitrogen	balance gas	139	158

Compound	Vapor LCS/LCSD (ppmv)	Water LCS/LCSD & MS/MSD	
		A	B
Hydrogen	25.00	126	----
Carbon Monoxide	-----	2.0	----

Notes on Table 4.2.2

- Actual values vary slightly from lot to lot of cylinders of calibration gases.
- MS/MSD prepared by using a standard gas mix instead of He in the headspace prep. procedure.

4.2.3.1 Total Inorganic Carbon MS and MSD

Mix approximately 0.04g NaHCO₃ directly into client samples (when provided and requested), prepare according to the TIC procedures outlined in PM01 and analyze in duplicate as a sample. The true value of the spike is calculated as follows:

$$\text{mg/L CaCO}_3 = \frac{\text{Mass}(g) \text{NaHCO}_3}{\text{H}_2\text{O}(L)} \times \frac{100.09}{84.01} \times (1,000,000)$$

4.2.4 Method Blank

Method blanks are made up of ultra high purity helium injected into a vial and then into the instrument.

4.2.4.1 Total Inorganic Carbon Method Blank

The method blank for TIC is made up of deionized water in a 40 ml vial, prepared according to the TIC procedures outlined in PM01, and analyzed as a sample.

4.3 Glassware and Storage Requirements for Reagents and Standards

Reagents are stored at room temperature (70°F ±5°) and all standards are prepared fresh for each use immediately prior to each analysis. Standards are made up from compressed gas cylinders. Those standards expire after 2 years.

5.0 Procedure

Water samples should be cooled upon collection and stored at a temperature of just above freezing but below 6°C.

Gas samples are shipped and received at a positive pressure, which eliminates a cross-contamination issue during sample shipment. It is preferable that gas samples be shipped without cooling. However, it is not a sample receipt non-conformance if received vapor samples are packed in ice (sample may experience slight loss in pressure.) Gas samples are stored in the laboratory at room temperature ($70^{\circ}\text{F} \pm 5^{\circ}$). The pressure in gas vials is not checked upon receipt in the laboratory because of the inherent risk of losing sample, or inadvertently introducing atmospheric gases, when the septum is pierced. The number of times the septum is pierced should be as few as absolutely possible. See Section 5.2.2 for a discussion on how the laboratory checks and documents vial pressure. Holding time for both gas and water samples is fourteen days.

Water samples for light hydrocarbon analyses only (methane, ethane, propane, propene, n-butane, i-butane, acetylene) are collected in 40ml VOA vials with zero headspace and preserved with tri-sodium phosphate (TSP). TSP is added as the dodecahydrate at 200 mg/40 ml vial. This results in a sample pH > 10. Water samples collected for either permanent gases only or permanent gases and light hydrocarbon analyses are collected in 40ml amber VOA vials with zero headspace and preserved with four drops of BAK solution. This solution acts as a microbial inhibitor and does not influence the buffered state of the sample.

Analysts who use this method have been certified for the method by running Initial Demonstration of Proficiency (IDOP) Samples in accordance with Microseeps Standard Operating Procedure for Administering and Documenting Training in Laboratory Procedures and Instrumentation (SOP ADM 02). IDOPs are run any time there is significant change to an instrument, method, or in the training procedure for training a new analyst.

Because the results from this method frequently require the analyst to use manual integration, manual integration is included as part of the training. Because of the nature of the instrument, the range of interrogated concentrations and the low specificity of the detectors, it is often necessary to perform manual integration even on the laboratory control samples. As part of the training, the analysts must:

- Retain an electronic copy of the original chromatogram that was integrated by the automated settings of the instrument software. (This is done automatically by the chromeleon software.)
- Document on the hard copy Case Narrative a justification for the manual integration and circle "YES" in the box in the lower right corner of the narrative sheet.
- The analyst shall present all the data for review.
- The reviewer shall thoroughly examine the data and when satisfied, check the appropriate box on the case narrative form and place their initials where designated.
- If there are questions about the manual integration, the data reviewer shall review the original chromatogram from the data system.
- If agreement is obtained from the data reviewer that the manual integration was indeed necessary, the reviewer shall document on the same hard copy Case Narrative (lower left

corner) that the manual integration was reviewed and the justification stands. If the other criteria of the training are met, the training is deemed successful.

- If the reviewer disagrees with either the necessity of the integration or the specific manipulations done in the integration, the specific objections should be discussed with the trainee and the training should be repeated (4 new samples must be analyzed).

5.1 Sample Preparation

Samples that are collected using the Bubble Strip Sampling Technique, Microseeps Sampling Method SM9, do not require additional preparation prior to analysis.

Samples that are collected as waters and are to be analyzed for dissolved gases (methane, ethane, ethene, acetylene, CO, CO₂, N₂, O₂, propane, propene, iso-butane, n-butane, TIC), must be prepared using Microseeps Standard Operating Procedure PM01G.

Samples that are collected as gases, for example from a soil gas survey or from the headspace of a microcosm sample, need not be collected by a Microseeps sampling method, nor do they require additional preparation.

5.2 Analysis

5.2.1 If the sample is prepared via SOP-PM 01, it can be injected from the syringe in which it is prepared by inserting the needle of the syringe through the septum on the "sample in" port. If the sample is a calibration standard, a bubble strip sample (SM9), or a gas, the septum inlet to the "sample in" port of the GC must be removed and a luer-lock needle receptacle is plumbed to the "sample in" port in place of the needle. A needle is attached to the luer-lock receptacle and inserted through the septa of the calibration standard, bubble stripped sample, or gas sample.

5.2.2 In order to initiate analysis and introduce the sample into the GC sample loop, a needle is attached to the entry port on the GC and inserted through the sample septum. The flow through the sample loop is monitored by a flow meter connected to the sample-loop vent-port on the gas chromatograph.

When a vial is sufficiently filled, the ball in the flow meter will shoot to the top of the column. This indicates that there is sufficient pressure in the vial to fill the sample loop. If the loop is not properly pressurized, this is reflected on the flow meter immediately. The ball in the flow meter will go up the column part way and drop back to the bottom. This indicates there is not sufficient pressure in the sample vial. If this happens, the analyst will remove the vial from the inlet port as quickly as possible and withdraw 10 – 12ccs of sample from the sample vial using a locking syringe. This is then injected into the instrument. The lack of sufficient pressure in the vial and the means of sample injection are then documented on the case narrative.

5.2.3 Once the flow out of the sample loop ceases (3 seconds if SOP-PM 01 is used) the sample loop valves are activated.

5.2.4 Once the sample loop valves are activated, the ports to and from the sample loop are flushed with ultra high purity helium injected into the loop directly from the cylinder to remove any interference from ambient air and to avoid cross contamination between samples.

5.3 Calibration and Results

5.3.1 The standard calibration gas should be introduced in the same manner as described in section 5.2.1 above. Measured peak areas are converted to concentrations using certified commercial gas standards. Dilutions are made to achieve multi-point calibration curves for each detector.

5.3.2 Initial calibration is accomplished by analyzing multiple standards of appropriate calibration ranges.

Note: Due to the nature of preparing custom gas standards, the component concentration can fluctuate between purchased lots. This is accounted for during method/calibration development. These results will be used to establish a multi-point calibration curve.

Acceptance Criteria: A linear fit to an area response versus concentration plot is formed with the origin forced to zero, and the calibration is accepted for use if r^2 , the coefficient of determination is ≥ 0.995 . If this criterion can not be met using a linear fit, a quadratic can be used. For the quadratic fit, the acceptance criteria is also $r^2 \geq 0.995$.

Corrective Action: If the acceptance criteria specified above is not met, the reason is determined and a new set of calibration standards are analyzed.

5.3.3 An Initial Calibration Verification (ICV) standard immediately follows the initial calibration. Acceptance criterion for the ICV is an instrument response within $\pm 15\%$ drift. Since the instrumentation used at Microseeps routinely monitors the percent recoveries and in this instance percent drift is equal to percent recovery less 100%, the control limits are 85%-115% recovery for the ICV. The ICV source shall be a second source, from a source other than those used for the calibration.

$$\text{Percent Recovery} = \frac{\text{Measured Value}}{\text{True Value}} \times 100\%$$

Acceptance Criteria and Corrective Action: If the instrument response for the ICV standard is outside the acceptance window of 85-115%, the analyst will not analyze samples until either the reason is determined and the problem is corrected, or a new multi-point calibration is analyzed and an acceptable ICV is run using that calibration.

5.3.4 An initial calibration blank follows the ICB. The blank is made up of the carrier gas. Compounds must not be detected above the reporting limits. For DoD projects the results of the ICB must be $< \frac{1}{2}$ RL.

Corrective Action: If the blank does not meet the acceptance criterion, another blank is injected until the results are within the acceptance criterion.

5.3.5 The analytes of this method are indicators. Every attempt to achieve and deliver precise results is made. However, it is realized that for indicator parameters measuring the range of the analyte concentration (*i.e.* is the concentration of methane gas >1 mg/l or < 0.1 mg/l) is the primary goal of employing these analyses. The calibration range is chosen to extend over most of the bio-indicator concentration range. If the concentration of an analyte exceeds that of the highest calibration standard, but does not saturate the instrument response, the concentration is calculated by assuming detector response linearity and using an extrapolation of the calibration plot. If the instrument response is saturated the sample is diluted to bring the analyte concentration into the calibration range.

5.4 Quality Control

The following quality control samples shall be analyzed with each analytical batch of fifteen or fewer samples.

5.4.1 A Continuing Calibration Verification: The CCV is made up from a source other than what was used to make up the initial calibration. The acceptance criterion for the CCV is a percent recovery of 85-115%. The CCV is also analyzed at the beginning and end of each analytical shift and after every 15 samples.

Corrective Action: If the CCV fails, a new CCV is prepared and analyzed. If the new CCV falls within the acceptance criterion, analysis continues. If the new CCV fails, the instrument shall be recalibrated, and all samples since the last acceptable calibration verification shall be reanalyzed, provided sufficient sample volume is present and the samples have not been compromised by exposure to air.

5.4.2 A Continuing Calibration Blank: A CCB follows each CCV. The blanks are made up of the carrier gas. The acceptance criterion for the blank is the result must be less than the reporting limits for all compounds. For DoD projects the results for the CCB must be $< \frac{1}{2}$ RL.

Corrective Action: If the blank does not meet the acceptance criterion, another blank is injected until the results are within the acceptance criterion.

5.4.3 Laboratory Control Sample and Laboratory Control Sample Duplicate: The LCS and LCSD are prepared and analyzed at a mid-calibration range, as defined in Table 4.2.2. Both an LCS and an LCSD are to be run with each batch. Water that is free of the principle atmospheric components of nitrogen and oxygen is very difficult to make and similarly difficult

to store. Toward that end, LCS/LCSD results for nitrogen and oxygen will be reported on a preliminary basis until procedural effectiveness is assured.

Note: Deionized source water for LCS/LCSD should be “degassed” prior to the addition of the source headspace.

Acceptance Criteria: Percent recovery is required to be between 80-120%, inclusive. An acceptance criterion is based upon the percent recovery and the RPD as calculated by:

$$\text{Percent Recovery} = \frac{\text{Measured Value}}{\text{True Value}} \times 100\%$$

$$\text{RPD} = \frac{|C1 - C2|}{\frac{C1 + C2}{2}} \times 100\%$$

Where: C1=LCS
C2=LCSD

RPD (Relative Percent Difference) is required to be less than or equal to 20%.

Corrective Action: If the LCS fails, a new LCS is prepared and analyzed. If the new LCS fails within the acceptance criterion, analysis continues. If the new LCS fails, analysis is stopped and the instrument is checked with a series of standards to determine the cause. Once the cause is determined and the instrument repaired, calibration is conducted and analysis continues.

5.4.4 Matrix Spike and Matrix Spike Duplicate: Matrix spikes and spike duplicates are analyzed for water samples only when requested by a client and sufficient sample aliquots are provided. Acceptance criterion is a percent recovery between 70% and 130% and a relative percent difference (RPD) of less than or equal to 20%.

The source used for MS/MSD will be based upon the methane concentration of the original field sample being spiked. Source concentration shall come from Table 4.2.2.

Corrective Action: If the matrix spike and spike duplicate fail but all the other quality control samples are within the acceptance criteria, matrix interference is noted in the Case Narrative.

5.4.5 Method Blank: A method blank is analyzed with each sample batch. The blanks are made up of UHP helium for all of the gases except for blanks for TIC. TIC blanks are made up of deionized water. The acceptance criterion for the blank is the result must be less than the reporting limits for all compounds. For DoD projects the results for the method blank must be < ½ RL.

Corrective Action: If the blank does not meet the acceptance criterion, another blank is injected until the results are within the acceptance criterion.

5.4.6 Contingency for Handling Out of Control or Unacceptable Data

If the requirements set forth in section 5.4 are not met, the analytical program will be terminated until the cause is determined and a solution is affected. All samples associated with out of control quality control samples (with the exception of matrix interference) must be reanalyzed provided another vial of sample has been provided by the client. If quality control acceptance criteria cannot be met using the corrective action above, a detailed check of the analytical system is made. Reagents, standards, and other quality control samples are re-prepared and analyzed. If problems persist, sample analysis will be halted and the Laboratory Manager shall be contacted immediately to determine the cause and implement corrective action.

Any data submitted with unacceptable quality control sample results shall be qualified in a case narrative. The narrative should indicate the out of control event that occurred, the corrective action that was taken, and any other pertinent information to inform the client of exactly what occurred.

5.4.7 An experienced analyst shall examine all chromatograms.

5.4.8 Through out analysis the gas samples are injected mechanically into the GC flow path utilizing a sample loop to achieve a uniform sample size from a flow directly from the sample preparation syringe. The uniform sample size achieved using the sample loop assures consistent and accurate results. Table 5.4.8 (see next page) gives example data from a study performed via this analysis. That data can also be used for accuracy and precision estimates.

Table 5.4.8
Example Data for Precision and Accuracy Studies

	Carbon Dioxide	Oxygen	Nitrogen	Methane	Hydrogen	Methane	Ethane	Ethylene	Propane	Propylene	Iso-Butane	N-Butane
REP. #	(% v)	(% v)	(% v)	(% v)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)
1	0.1221	0.0670	0.5744	0.0410	0.1118	0.2512	0.0525	0.0453	0.0461	0.0581	0.0473	0.0358
2	0.1267	0.0690	0.6020	0.0428	0.1122	0.2608	0.0518	0.0468	0.0521	0.0465	0.0439	0.0407
3	0.1207	0.0657	0.5838	0.0446	0.1247	0.2812	0.0509	0.0485	0.0529	0.0588	0.0436	0.0405
4	0.1193	0.0667	0.6036	0.0444	0.1244	0.2779	0.0549	0.0460	0.0461	0.0536	0.0549	0.0476
5	0.1261	0.0703	0.5860	0.0439	0.1120	0.2894	0.0551	0.0497	0.0520	0.0549	0.0417	0.0460
6	0.1193	0.0665	0.5861	0.0478	0.0943	0.2970	0.0515	0.0467	0.0458	0.0542	0.0435	0.0514
7	0.1227	0.0732	0.5748	0.0353	0.1296	0.3053	0.0532	0.0473	0.0485	0.0584	0.0483	0.0535
AVERAGE	0.1224	0.0683	0.5872	0.0428	0.1156	0.2804	0.0528	0.0472	0.0491	0.0549	0.0462	0.0451
KNOWN	0.1500	0.0700	0.6649	0.0450	0.0999	0.1500	0.0500	0.0500	0.0500	0.0500	0.0500	0.0500
STD. DEV.	0.003	0.003	0.012	0.004	0.012	0.019	0.002	0.001	0.003	0.004	0.004	0.006

5.4.9 The gas matrix for this analysis minimizes the opportunity for matrix effects. If the gas is prepared from a matrix other than that which is injected into the GC (*e.g.* prepared through headspace extraction via Microseeps SOP-PM01), the client should request that matrix spike (MS) and matrix spike duplicate (MSD) analyses be conducted and should supply sufficient sample volume. Since matrix effects are extremely site dependent, the MS and MSD are not part of the regular analytical quality assurance program.

5.4.10 All of the target analytes gases are at room temperature so the opportunity for carry over is small. This is further reduced by the flushing of the sample loop, by the “backflush” configuration of the GC plumbing, and by the nightly bake-out procedure. These combine to keep carry-over concentrations to less than half of the reporting limits.

5.5 Capturing and Submitting Data

The output of the chromatograph is directed to a microcomputer where the signal is converted to digital format, stored, and processed using a chromatography data system.

Automated valve control: Digital control is provided by the microcomputer through the chromatography data-system software. This control provides constant start and stop times for directing carrier gas flow. The event times are programmed and saved using the method editor module of the software.

5.5.1 Total Inorganic Carbon Result Calculation

The total inorganic carbon result is calculated as follows:

$$\text{TIC as mg/L CaCO}_3 = (\% \text{CO}_2)((\text{Volume headspace})(2.08) + 43.3)$$

This analysis produces concentration of the analyzed gas in % V.

5.5.2 Retention Time Windows

Retention time studies have been conducted for this analysis. These studies are kept on file in the Quality Systems Office. The exact retention times will vary as a function of column type, column age, and column history. For the instruments that use this method, true retention times and retention time windows are taken from the most recent standard analyzed.

5.5.2.1 Determination of Retention Time Windows

Inject a standard a total of 3 times over a 72 hour period. Record the retention time for each component to a minimum of 3 decimal places. Calculate the mean and standard deviation of the three absolute retention times for each component. If the standard deviation for a target compound is 0.000, use a default standard deviation of 0.01 minutes. The width of the retention time window for each analyte is ± 3 times the standard deviation of the mean absolute retention

time. If the default standard deviation 0.01 minutes is used, the width of the window will be 0.03 minutes.

Establish the center of the retention time window for each analyte by using the absolute retention time for each analyte from the calibration verification standard at the beginning of the batch.

Retention time windows must be calculated for each instrument and column used. New retention time windows must be established when a new column is installed.

5.6 Bake-out Procedure

Either overnight, through the weekend or whenever the instrument is not going to be used for several hours, the instrument is put in “bake-out”. With carrier gas continuous flushing through the GC, the temperature on the oven is manually turned up to 210 degrees or as high as the instrument column oven can maintain.

6.0 Secondary Data Review

All analytical data must undergo a minimum of a two-tiered review. The analyst first reviews the data for completeness and accuracy. The data is then submitted to the Group Lead Analyst for final review and the data is entered into the LIMS. Once approved at this level, the data is uploaded into the LIMS.

7.0 Reporting Limits

The reporting limits for this analysis are listed in Table 7.0 below. Method detection limit studies are run annually in accordance with Microseeps Standard Operating Procedure for the Determination of Method Detection Limits and PQLs (SOP-ADM 18).

Those MDLs must be less than the reporting limits specified below. MDL studies are also performed when there is reason to suspect that method sensitivity has changed. The MDL studies are kept on file in the Quality Systems Office.

Reporting Limits

Table 7.0

Parameter	Reporting Limit	Units
Carbon Monoxide	0.20	% V
Carbon Dioxide	0.2	% V

Parameter	Reporting Limit	Units
Oxygen	0.2	% V
Nitrogen	0.5	% V
Hydrogen	0.5	ppmv
Acetylene	0.2	ppmv
Methane	0.2	ppmv
Ethane	0.1	ppmv
Ethene	0.1	ppmv
Propane	0.1	ppmv
Propene	0.2	ppmv
n-Butane	0.2	ppmv
i-Butane	0.2	ppmv

7.1 Conversion Factors

This procedure is used to measure the volume concentration of the analytes in a gas. Two methods are used to extract that gas from the groundwater. The conversion factors that are used to convert the concentration of the analytes in the water from the concentration of the analytes as they are measured using this method, are specific to the collection or preparation method and can be found in either SOP-SM9 or SOP-PM 01.

8.0 Safety

Gloves, proper eye protection, and a laboratory coat shall be worn when handling samples and standards. The major hazard in this laboratory area is stick from needles. All needles must be capped when not in use and when moving about the laboratory. The proper way of capping a needle is to place the cap on the laboratory bench and direct the needle into the cap. A needle is never to be directed into a cap while the cap is being held.

All compressed gases are to be moved using a dolly made for transporting gases and shall be chained in place when in the laboratory. The chain shall be tightened sufficiently to keep the cylinder upright if jostled.

Safety glasses are to be worn in all laboratory areas. Material Safety Data Sheets (MSDSs) for all compounds used in this procedure are available in the laboratory.

9.0 Laboratory Waste

Samples are kept for 30 days following analysis. Samples are disposed according to Microseeps Standard Operation Procedure for Waste Disposal (SOP-ADM 14).

9.1 Waste Minimization

Where possible, Microseeps takes steps to minimize the amount of waste generated in the laboratory by using substitution, where possible, and good chemical handling procedures. For specific information on waste minimization consult SOP-ADM 14.

10.0 References

Citing a reference does not imply that all of the recommendations and/or requirements in those cited methods is required in this Standard Operating Procedure. This section simply refers to sources that were consulted to gather information or knowledge in order to write an informed technical procedure.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste. SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, DC. 1986.

Newel, B.S., RSK-SOP-175, Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples using GC Headspace Equilibration Technique. Revision No. 0, August 1994.

American Society for Testing and Materials, Standard Practice for Analysis of Reformed Gas by Gas Chromatography. Annual Book of ASTM Standards. Vol. 14.02, 1994.

Kampbell, D.H. and Vandegrift, S.A., Analysis of Dissolved Methane, Ethane, and Ethylene in Ground Water by a Standard Gas Chromatographic Technique. Journal of Chromatographic Science. Vol. 36, May 1998.

SOP Review Date: September 5, 2013

Microseeps, Incorporated

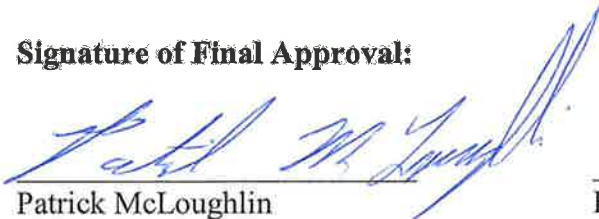
Standard Operating Procedure for Regular Equipment Maintenance

Controlled Copy _____



Mark Mikesell
Laboratory Manager

Signature of Final Approval:



Patrick McLoughlin
Technical Director

4/27/12
Effective Date

1.0 Introduction and Scope

Maintenance is performed on a regular, or as needed, basis to ensure that equipment is operating properly. Regular maintenance would be considered preventative and as needed maintenance would be considered for repair. The manual for each piece of equipment shall be kept in the laboratory in which the equipment is located. The equipment manuals shall be available to the analyst at all times.

The following pages outline regular maintenance requirements for all laboratory equipment and analytical instrumentation.

2.0 Responsibility

2.1 Instrumentation Specialist

The Instrumentation Specialist makes the determination whether maintenance shall be performed in-house, or whether it is necessary to bring in a vendor representative. The Instrumentation Specialist shall perform all in-house maintenance that cannot be done by the analyst. The Instrumentation Specialist shall also complete the bottom portion of the Maintenance Request Form (Exhibit 1) and submit a copy to the Laboratory Manager and the Quality Systems Coordinator immediately upon completion of the maintenance.

2.2 Laboratory Manager

The Laboratory Manager will coordinate with the Instrumentation Specialist once a Maintenance Request Form (Exhibit 1) is initiated and will communicate with Customer Care concerning any instrument outages that will or can affect project requirements/specifics and turnaround times. The Laboratory Manager will coordinate with the analyst any IDOP or MDL studies that require repeating, should maintenance suggest the need for such analyses.

2.3 Analyst

The Analysts are responsible for ensuring that the instruments are maintained in accordance with this Standard Operating Procedure. Analysts are responsible for assigning regular maintenance tasks and documenting their completion. When maintenance of an instrument is necessary that cannot be completed by the analyst, the analyst shall initiate a Maintenance Request Form (Exhibit 1) and submit the form to the Laboratory Manager. The Analysts are responsible for ensuring that repeat IDOPs and MDL studies are completed on the instrument when major changes are made to the instrument that affect the sensitivity prior to analyzing client samples.

NOTE: All maintenance shall be documented in the Instrument Maintenance Logbook.

2.4 Quality Systems Coordinator

Logbooks can be obtained from the Quality Systems Coordinator. The Quality Systems Coordinator shall notify the Laboratory Manager when repeat IDOP and MDL studies must be conducted due to any maintenance that affects instrument sensitivity.

3.0 Out of Service

Each laboratory has been issued tags for out of service instruments. These tags are to be placed on equipment that is out of service, and left on the instrument until it is brought back into service.

If at any time, the equipment is in need of service, place the "Out of Service" tag on the instrument, discontinue using it, complete the attached form (Exhibit 1), and submit it to the Laboratory Manager immediately. These forms will be submitted to the Instrumentation Specialist. The Instrumentation Specialist will determine if the maintenance required on the instrument or equipment prohibits the use of the equipment until repairs are made or if use can continue until a scheduled repair can be made.

4.0 Equipment Maintenance Documentation

Information that is required to be entered in the Equipment Maintenance Logbook is as follows:

- Name of item of equipment.
- Manufacturer's name.
- Identification/Serial number of equipment.
- Date equipment was received by the laboratory (if known).
- Date equipment was placed in service (if known).
- Current location (room number).
- Condition equipment was in when received (i.e. new, used, reconditioned).

5.0 Equipment Maintenance Procedures

The following codes shall be used to designate the frequency at which a particular maintenance procedure is to be performed.

D = Daily
W = Weekly
M = Monthly

Q = Quarterly

SA = Semi-annually

A = Annually

As Needed: Whenever contamination or problems are suspected.

SOP Review Date: November 18, 2011

Controlled Document

**Regular Maintenance Program
Gas Chromatography/Mass Spectrometer**

<u>Task</u>	<u>Frequency</u>
Check septa and replace, if necessary	D
Check cylinder gas pressure	D
Check and replace glass insert in injector body if necessary	D
Replace vacuum pump oil	A
Check ion source and analyzer (dismantle, clean, replace parts as needed)	As needed
Check mechanical systems (vacuum pumps, relays, gas pressure and flows)	A
Check mass calibration w/FC-43 (perfluorotributylamine)	As needed
Clean sparger tube	As needed*
Change Trap	As needed*
Bake Trap	D*
Replace syringe	As needed

*Denotes parts of the Concentrator for purge and trap systems.

**Regular Maintenance Program
Gas Chromatography**

<u>Task</u>	<u>Frequency</u>
Check septa and replace, if necessary.	D
Check gas cylinder pressure replace cylinder, if necessary.	D
Check and replace glass insert in injector body, if necessary.	D
Wipe tests on Electron capture detector.	A
Return ECD for cleaning.	As needed
Clean FID (jet, collector and insulators)	As needed
Replace filaments on TCD (replace in pairs)	As needed
Change Activated Carbon (RGD detector)	SA
Bake out Columns	D

**Regular Maintenance Program
For Other Laboratory Instrumentation and Equipment**

<u>Task</u>	<u>Frequency</u>
TOC Analyzer	
Check gas cylinder/pressure replace if necessary	D
Check persulfate, acid, and DI water supply	D
Check chlorine scrubber replace packing if needed	D
Gas/Liquid Separator water level filled to waste outlet	D
Clean UV Reactor and IC Sparger with soap & water if needed	As needed
Replace water in Gas/Liquid Separator	As needed
UV/Vis Spectrophotometer	
Windows cleaned	M
Check and adjust photo-multiplier sensitivity and wavelength resolution	A
Replace lamp	As needed
Clean sample compartment before and after use	D
Clean cuvettes after each use	D
pH and Ion Selective Electrodes	
Probe: Check for cracks, filling solution; check reference junction; clean electrode replace if necessary	D
Check junctions and clean if necessary	D
Meter: Check batteries, electronics for loose connections or cracked leads	D

**Regular Maintenance Program
For Other Laboratory Instrumentation and Equipment Continued**

<u>Task</u>	<u>Frequency</u>
Ion Chromatograph:	
Cation Side:	
Prepare Reagents	As needed
De-gas water/reagents	As needed
Prime pumps	D
Check gas cylinder/replace if necessary	D
Check flow	D
Anion Side:	
Prime pumps	As needed
Check gas cylinder/replace if necessary	D
Check conductivity	D
Flush analytical column	As needed
Replace end filters	As needed
Clean check valves	As needed
Flush PEEK line	As needed

Regular Maintenance Program For Other Laboratory Instrumentation and Equipment Continued

Refrigerators, and Ovens:

Clean interior	M
Defrost (if applicable)	Q
Check Temperature (report to QC if out of spec.)	D

Balances:

Clean pan and compartment	D
Check with class S weights	D
Manufacturer or Service Contract (cleaning and calibration)	A

Deionized Water System:

Purelab Prima	
Replace LC123 Pre-treatment filter	SA
Replace LC140 Pre-treatment filter	SA
Replace LC143 Reverse Osmosis filter	2-3 years
Replace LC136 Composite Vent filter	SA
Purelab Ultra	
Replace LC182 Labpure SI Primary Purification Filter Pack	SA
Replace LC118 Ultraviolet Lamp/Chamber	A
Replace LC182 Labpure SI Polishing Purification Filter Pack	SA
Replace LC109 Ultra Microfiber filter	SA

Note: The above schedule is that suggested by the manufacturer, however, the laboratory has determined that 24-36 month time frame is adequate, based on the units performance to date.

Maintenance Request Form

ATTN: **Laboratory Manager**

Date: _____

Room Number: _____ Analyst: _____

Equipment: _____

Describe the problem: _____

(Bottom portion of form to be completed by Instrumentation Specialist)

Maintenance to be done by: _____ In house

_____ Vendor

Vendor Name: _____

Date Contacted: _____

Date Set for Service Call: _____

In-house Maintenance Date: _____ Conducted by: _____

This maintenance procedure did ____ did not ____ require a change or modification of the equipment that is considered major (a new column or other change that affects instrument sensitivity.)

cc: Completed form to Analyst and Quality Department.

Appendix E
Laboratory Control Charts

Printed: 07/13/2013 08:16

Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

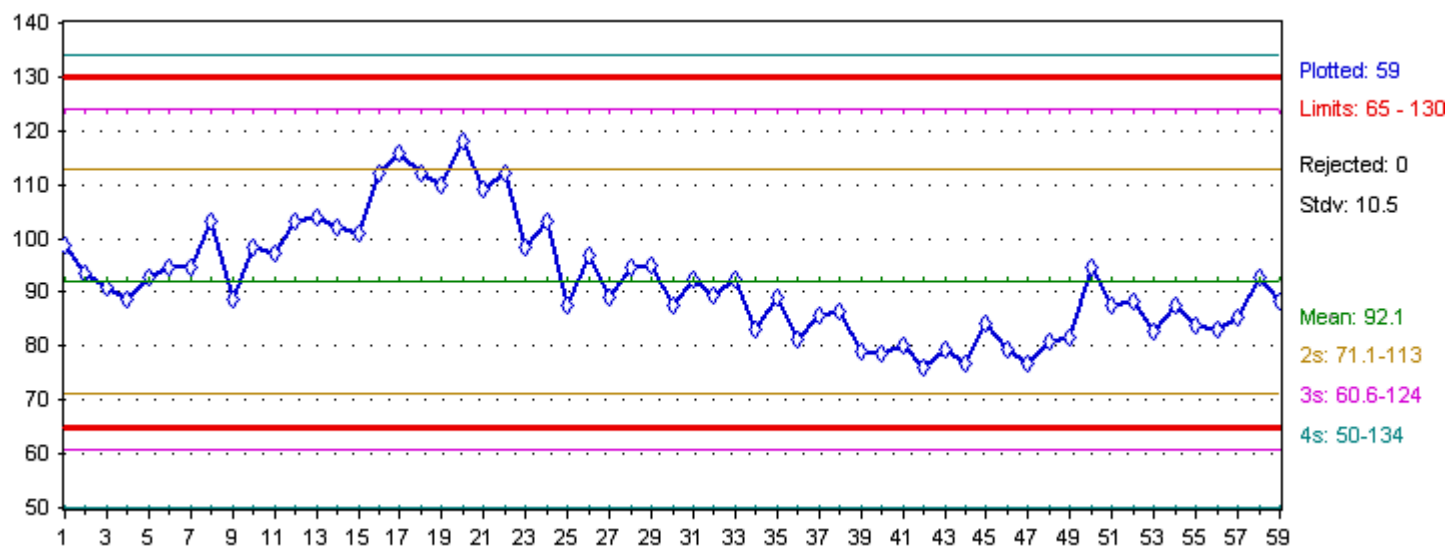
Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

1,1,2,2-Tetrachloroethane

LCS %R



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3D15001-BS1	4/15/13	4/15/13	50	49.3	98.58	65-130	
	3D16001-BS1	4/16/13	4/16/13	50	46.8	93.5	65-130	
	3D17005-BS1	4/17/13	4/17/13	50	45.4	90.78	65-130	
	3D18016-BS1	4/18/13	4/18/13	50	44.3	88.66	65-130	
	3D19006-BS1	4/19/13	4/19/13	50	46.5	92.92	65-130	
	3D22004-BS1	4/22/13	4/22/13	50	47.4	94.72	65-130	
	3D23008-BS1	4/23/13	4/23/13	50	47.3	94.66	65-130	
	3D24009-BS1	4/24/13	4/24/13	50	51.3	102.62	65-130	
	3D25008-BS1	4/25/13	4/25/13	50	44.4	88.74	65-130	
	3D26005-BS1	4/26/13	4/26/13	50	49.2	98.46	65-130	
	3D29008-BS1	4/29/13	4/29/13	50	48.7	97.42	65-130	
	3D30017-BS1	4/30/13	4/30/13	50	51.3	102.52	65-130	
	3D30025-BS1	4/30/13	4/30/13	50	51.8	103.66	65-130	
	3E01005-BS1	5/1/13	5/1/13	50	51	101.98	65-130	
	3E06002-BS1	5/6/13	5/6/13	50	50.3	100.58	65-130	
	3E07006-BS1	5/7/13	5/7/13	50	56	111.9	65-130	
	3E08004-BS1	5/8/13	5/8/13	50	57.8	115.64	65-130	
	3E08018-BS1	5/8/13	5/8/13	50	55.8	111.58	65-130	
	3E09011-BS1	5/9/13	5/9/13	50	54.8	109.66	65-130	
	3E10010-BS1	5/10/13	5/10/13	50	59.1	118.26	65-130	
	3E11006-BS1	5/11/13	5/11/13	50	54.7	109.36	65-130	
	3E13317-BS1	5/13/13	5/13/13	50	55.9	111.8	65-130	
	3E14013-BS1	5/14/13	5/14/13	50	49.3	98.54	65-130	
	3E14018-BS1	5/14/13	5/14/13	50	51.6	103.24	65-130	
	3E15010-BS1	5/15/13	5/15/13	50	43.9	87.72	65-130	
	3E16019-BS1	5/16/13	5/16/13	50	48.3	96.66	65-130	
	3E17010-BS1	5/17/13	5/17/13	50	44.5	88.96	65-130	

Printed: 07/13/2013 08:16

Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

1,1,2,2-Tetrachloroethane					LCS %R		Limits	Qualifiers
Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R		
	3E17016-BS1	5/17/13	5/17/13	50	47.3	94.64	65-130	
	3E18005-BS1	5/18/13	5/18/13	50	47.5	95.02	65-130	
	3E20006-BS1	5/20/13	5/20/13	50	43.8	87.52	65-130	
	3E20018-BS1	5/20/13	5/20/13	50	46.2	92.44	65-130	
	3E21009-BS1	5/21/13	5/21/13	50	44.7	89.38	65-130	
	3E21026-BS1	5/21/13	5/21/13	50	46.2	92.32	65-130	
	3E22016-BS1	5/22/13	5/22/13	50	41.5	83.02	65-130	
	3E22027-BS1	5/22/13	5/22/13	50	44.5	88.94	65-130	
	3E23011-BS1	5/23/13	5/23/13	50	40.7	81.34	65-130	
	3E23019-BS1	5/23/13	5/23/13	50	42.9	85.76	65-130	
	3E24013-BS1	5/24/13	5/24/13	50	43.2	86.4	65-130	
	3E24019-BS1	5/24/13	5/24/13	50	39.5	78.94	65-130	
	3E25004-BS1	5/25/13	5/25/13	50	39.3	78.58	65-130	
	3E25008-BS1	5/26/13	5/26/13	50	40	80.04	65-130	
	3E28013-BS1	5/28/13	5/28/13	50	38	76.04	65-130	
	3E28023-BS1	5/28/13	5/28/13	50	39.8	79.52	65-130	
	3E29016-BS1	5/29/13	5/29/13	50	38.4	76.9	65-130	
	3E30015-BS1	5/30/13	5/30/13	50	42	84.06	65-130	
	3E31003-BS1	5/31/13	5/31/13	50	39.7	79.42	65-130	
	3E31016-BS1	5/31/13	5/31/13	50	38.4	76.8	65-130	
	3F01002-BS1	6/1/13	6/1/13	50	40.3	80.68	65-130	
	3F01003-BS1	6/1/13	6/1/13	50	40.8	81.62	65-130	
	3F03022-BS1	6/3/13	6/3/13	50	47.3	94.6	65-130	
	3F04012-BS1	6/4/13	6/4/13	50	43.8	87.7	65-130	
	3F04013-BS1	6/4/13	6/4/13	50	44.1	88.26	65-130	
	3F05001-BS1	6/5/13	6/5/13	50	41.3	82.6	65-130	
	3F05018-BS1	6/5/13	6/5/13	50	43.7	87.44	65-130	
	3F06010-BS1	6/6/13	6/6/13	50	41.9	83.8	65-130	
	3F07001-BS1	6/7/13	6/7/13	50	41.5	83.02	65-130	
	3F07034-BS1	6/7/13	6/7/13	50	42.7	85.48	65-130	
	3F13017-BS1	6/13/13	6/13/13	50	46.3	92.58	65-130	
	3F13024-BS1	6/13/13	6/13/13	50	44.1	88.24	65-130	

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Matrices: Water

Client: All Clients

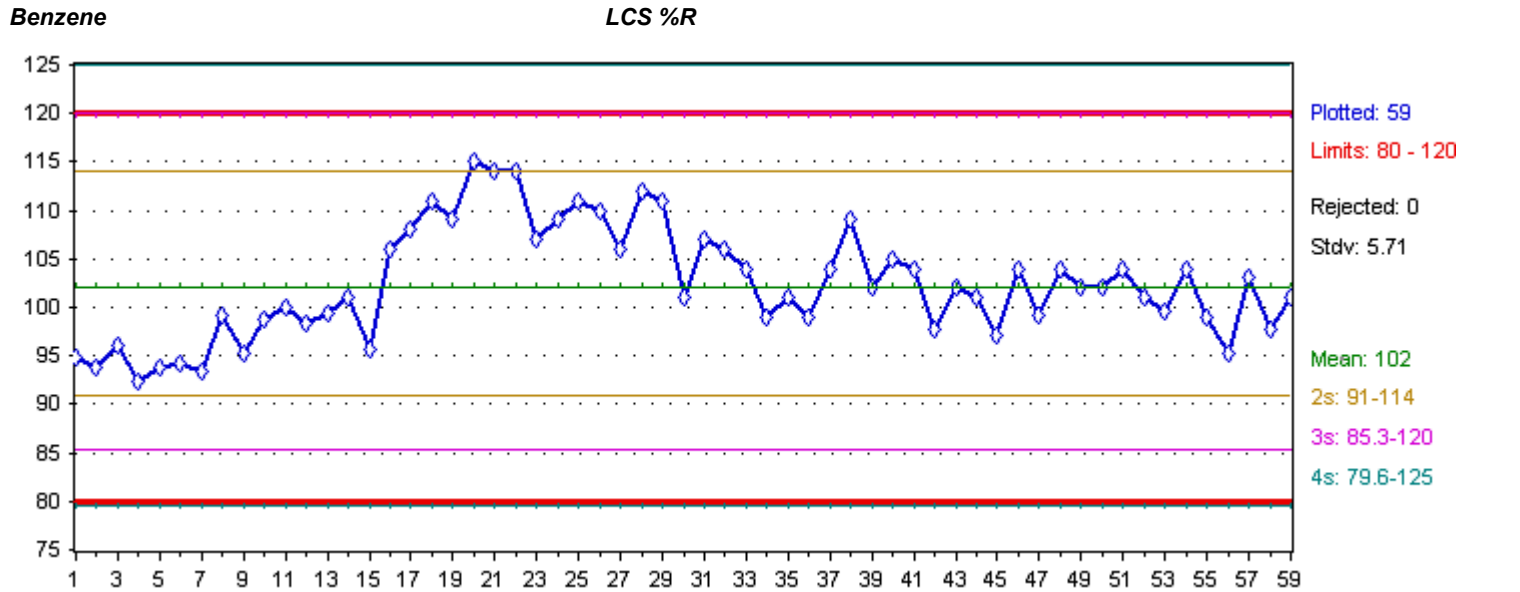
Instruments: gcmsvoa5

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3D15001-BS1	4/15/13	4/15/13	50	47.4	94.88	80-120	
	3D16001-BS1	4/16/13	4/16/13	50	46.9	93.74	80-120	
	3D17005-BS1	4/17/13	4/17/13	50	48.1	96.12	80-120	
	3D18016-BS1	4/18/13	4/18/13	50	46.2	92.38	80-120	
	3D19006-BS1	4/19/13	4/19/13	50	46.9	93.78	80-120	
	3D22004-BS1	4/22/13	4/22/13	50	47.2	94.3	80-120	
	3D23008-BS1	4/23/13	4/23/13	50	46.7	93.42	80-120	
	3D24009-BS1	4/24/13	4/24/13	50	49.6	99.1	80-120	
	3D25008-BS1	4/25/13	4/25/13	50	47.6	95.3	80-120	
	3D26005-BS1	4/26/13	4/26/13	50	49.4	98.74	80-120	
	3D29008-BS1	4/29/13	4/29/13	50	50.1	100.28	80-120	
	3D30017-BS1	4/30/13	4/30/13	50	49.2	98.3	80-120	
	3D30025-BS1	4/30/13	4/30/13	50	49.7	99.34	80-120	
	3E01005-BS1	5/1/13	5/1/13	50	50.3	100.56	80-120	
	3E06002-BS1	5/6/13	5/6/13	50	47.9	95.74	80-120	
	3E07006-BS1	5/7/13	5/7/13	50	53.2	106.38	80-120	
	3E08004-BS1	5/8/13	5/8/13	50	54.2	108.4	80-120	
	3E08018-BS1	5/8/13	5/8/13	50	55.4	110.72	80-120	
	3E09011-BS1	5/9/13	5/9/13	50	54.5	108.98	80-120	
	3E10010-BS1	5/10/13	5/10/13	50	57.4	114.82	80-120	
	3E11006-BS1	5/11/13	5/11/13	50	57.2	114.3	80-120	
	3E13317-BS1	5/13/13	5/13/13	50	56.9	113.86	80-120	
	3E14013-BS1	5/14/13	5/14/13	50	53.6	107.12	80-120	
	3E14018-BS1	5/14/13	5/14/13	50	54.6	109.18	80-120	
	3E15010-BS1	5/15/13	5/15/13	50	55.4	110.74	80-120	
	3E16019-BS1	5/16/13	5/16/13	50	55.2	110.36	80-120	
	3E17010-BS1	5/17/13	5/17/13	50	53.1	106.28	80-120	

Printed: 07/13/2013 08:16

Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

Benzene		LCS %R					Limits	Qualifiers
Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R		
	3E17016-BS1	5/17/13	5/17/13	50	55.9	111.74	80-120	
	3E18005-BS1	5/18/13	5/18/13	50	55.4	110.78	80-120	
	3E20006-BS1	5/20/13	5/20/13	50	50.6	101.2	80-120	
	3E20018-BS1	5/20/13	5/20/13	50	53.5	107.02	80-120	
	3E21009-BS1	5/21/13	5/21/13	50	53.2	106.36	80-120	
	3E21026-BS1	5/21/13	5/21/13	50	52.2	104.38	80-120	
	3E22016-BS1	5/22/13	5/22/13	50	49.5	98.98	80-120	
	3E22027-BS1	5/22/13	5/22/13	50	50.7	101.48	80-120	
	3E23011-BS1	5/23/13	5/23/13	50	49.5	99.04	80-120	
	3E23019-BS1	5/23/13	5/23/13	50	51.9	103.72	80-120	
	3E24013-BS1	5/24/13	5/24/13	50	54.3	108.68	80-120	
	3E24019-BS1	5/24/13	5/24/13	50	50.9	101.78	80-120	
	3E25004-BS1	5/25/13	5/25/13	50	52.6	105.1	80-120	
	3E25008-BS1	5/26/13	5/26/13	50	52	103.98	80-120	
	3E28013-BS1	5/28/13	5/28/13	50	48.9	97.72	80-120	
	3E28023-BS1	5/28/13	5/28/13	50	51.1	102.22	80-120	
	3E29016-BS1	5/29/13	5/29/13	50	50.5	101.02	80-120	
	3E30015-BS1	5/30/13	5/30/13	50	48.6	97.16	80-120	
	3E31003-BS1	5/31/13	5/31/13	50	51.8	103.5	80-120	
	3E31016-BS1	5/31/13	5/31/13	50	49.6	99.16	80-120	
	3F01002-BS1	6/1/13	6/1/13	50	51.9	103.86	80-120	
	3F01003-BS1	6/1/13	6/1/13	50	51	101.92	80-120	
	3F03022-BS1	6/3/13	6/3/13	50	51.1	102.2	80-120	
	3F04012-BS1	6/4/13	6/4/13	50	52	104.04	80-120	
	3F04013-BS1	6/4/13	6/4/13	50	50.4	100.76	80-120	
	3F05001-BS1	6/5/13	6/5/13	50	49.8	99.58	80-120	
	3F05018-BS1	6/5/13	6/5/13	50	52.2	104.38	80-120	
	3F06010-BS1	6/6/13	6/6/13	50	49.5	99.02	80-120	
	3F07001-BS1	6/7/13	6/7/13	50	47.7	95.34	80-120	
	3F07034-BS1	6/7/13	6/7/13	50	51.3	102.56	80-120	
	3F13017-BS1	6/13/13	6/13/13	50	48.9	97.76	80-120	
	3F13024-BS1	6/13/13	6/13/13	50	50.7	101.32	80-120	

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Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

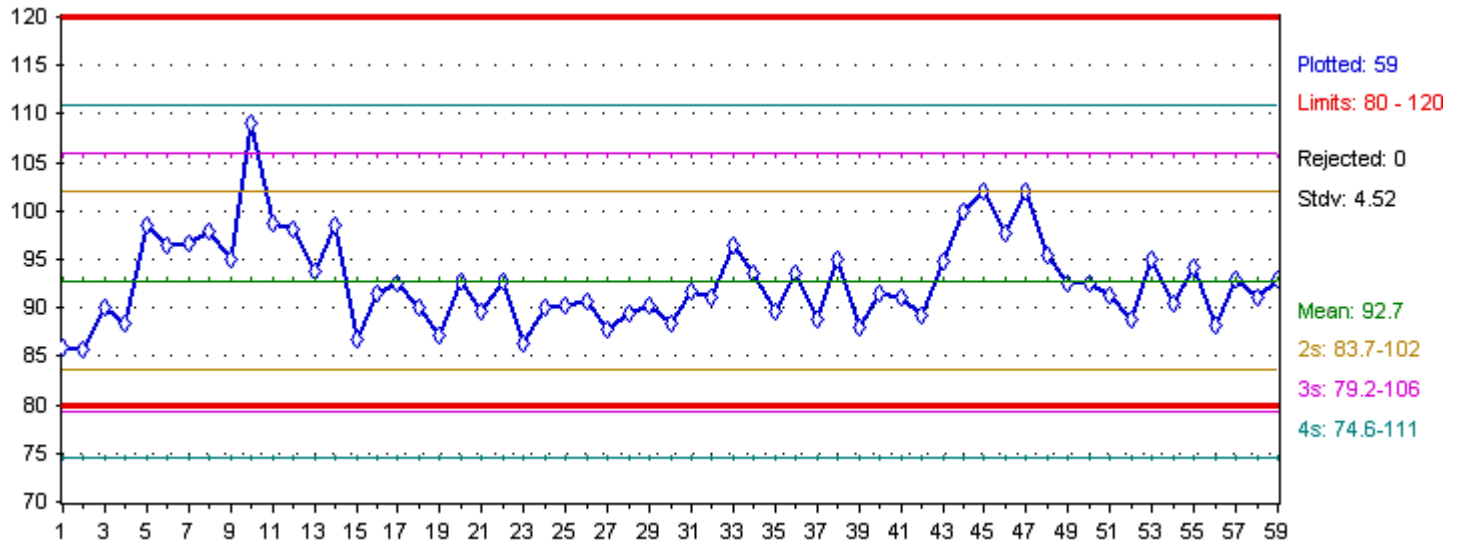
Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

Chlorobenzene

LCS %R



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3D15001-BS1	4/15/13	4/15/13	50	43	85.92	80-120	
	3D16001-BS1	4/16/13	4/16/13	50	42.9	85.76	80-120	
	3D17005-BS1	4/17/13	4/17/13	50	45	90.1	80-120	
	3D18016-BS1	4/18/13	4/18/13	50	44.1	88.26	80-120	
	3D19006-BS1	4/19/13	4/19/13	50	49.3	98.58	80-120	
	3D22004-BS1	4/22/13	4/22/13	50	48.3	96.52	80-120	
	3D23008-BS1	4/23/13	4/23/13	50	48.3	96.68	80-120	
	3D24009-BS1	4/24/13	4/24/13	50	48.9	97.88	80-120	
	3D25008-BS1	4/25/13	4/25/13	50	47.5	95.08	80-120	
	3D26005-BS1	4/26/13	4/26/13	50	54.4	108.88	80-120	
	3D29008-BS1	4/29/13	4/29/13	50	49.4	98.72	80-120	
	3D30017-BS1	4/30/13	4/30/13	50	49	98.1	80-120	
	3D30025-BS1	4/30/13	4/30/13	50	46.9	93.84	80-120	
	3E01005-BS1	5/1/13	5/1/13	50	49.3	98.54	80-120	
	3E06002-BS1	5/6/13	5/6/13	50	43.4	86.74	80-120	
	3E07006-BS1	5/7/13	5/7/13	50	45.7	91.38	80-120	
	3E08004-BS1	5/8/13	5/8/13	50	46.3	92.64	80-120	
	3E08018-BS1	5/8/13	5/8/13	50	45	90.06	80-120	
	3E09011-BS1	5/9/13	5/9/13	50	43.6	87.16	80-120	
	3E10010-BS1	5/10/13	5/10/13	50	46.4	92.82	80-120	
	3E11006-BS1	5/11/13	5/11/13	50	44.8	89.6	80-120	
	3E13317-BS1	5/13/13	5/13/13	50	46.4	92.82	80-120	
	3E14013-BS1	5/14/13	5/14/13	50	43.2	86.36	80-120	
	3E14018-BS1	5/14/13	5/14/13	50	45	90.02	80-120	
	3E15010-BS1	5/15/13	5/15/13	50	45.2	90.32	80-120	
	3E16019-BS1	5/16/13	5/16/13	50	45.4	90.72	80-120	
	3E17010-BS1	5/17/13	5/17/13	50	43.9	87.8	80-120	

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Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

Chlorobenzene			LCS %R				Limits	Qualifiers
Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R		
	3E17016-BS1	5/17/13	5/17/13	50	44.8	89.52	80-120	
	3E18005-BS1	5/18/13	5/18/13	50	45.1	90.28	80-120	
	3E20006-BS1	5/20/13	5/20/13	50	44.2	88.38	80-120	
	3E20018-BS1	5/20/13	5/20/13	50	45.8	91.56	80-120	
	3E21009-BS1	5/21/13	5/21/13	50	45.5	91.08	80-120	
	3E21026-BS1	5/21/13	5/21/13	50	48.2	96.38	80-120	
	3E22016-BS1	5/22/13	5/22/13	50	46.8	93.56	80-120	
	3E22027-BS1	5/22/13	5/22/13	50	44.8	89.62	80-120	
	3E23011-BS1	5/23/13	5/23/13	50	46.8	93.52	80-120	
	3E23019-BS1	5/23/13	5/23/13	50	44.3	88.68	80-120	
	3E24013-BS1	5/24/13	5/24/13	50	47.4	94.88	80-120	
	3E24019-BS1	5/24/13	5/24/13	50	44	88.04	80-120	
	3E25004-BS1	5/25/13	5/25/13	50	45.7	91.44	80-120	
	3E25008-BS1	5/26/13	5/26/13	50	45.5	91.08	80-120	
	3E28013-BS1	5/28/13	5/28/13	50	44.6	89.24	80-120	
	3E28023-BS1	5/28/13	5/28/13	50	47.4	94.72	80-120	
	3E29016-BS1	5/29/13	5/29/13	50	50	100.04	80-120	
	3E30015-BS1	5/30/13	5/30/13	50	51	101.94	80-120	
	3E31003-BS1	5/31/13	5/31/13	50	48.9	97.74	80-120	
	3E31016-BS1	5/31/13	5/31/13	50	51.1	102.14	80-120	
	3F01002-BS1	6/1/13	6/1/13	50	47.7	95.36	80-120	
	3F01003-BS1	6/1/13	6/1/13	50	46.2	92.5	80-120	
	3F03022-BS1	6/3/13	6/3/13	50	46.3	92.64	80-120	
	3F04012-BS1	6/4/13	6/4/13	50	45.6	91.2	80-120	
	3F04013-BS1	6/4/13	6/4/13	50	44.4	88.88	80-120	
	3F05001-BS1	6/5/13	6/5/13	50	47.5	94.94	80-120	
	3F05018-BS1	6/5/13	6/5/13	50	45.2	90.42	80-120	
	3F06010-BS1	6/6/13	6/6/13	50	47.1	94.16	80-120	
	3F07001-BS1	6/7/13	6/7/13	50	44.1	88.14	80-120	
	3F07034-BS1	6/7/13	6/7/13	50	46.5	93.04	80-120	
	3F13017-BS1	6/13/13	6/13/13	50	45.6	91.1	80-120	
	3F13024-BS1	6/13/13	6/13/13	50	46.5	92.96	80-120	

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Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

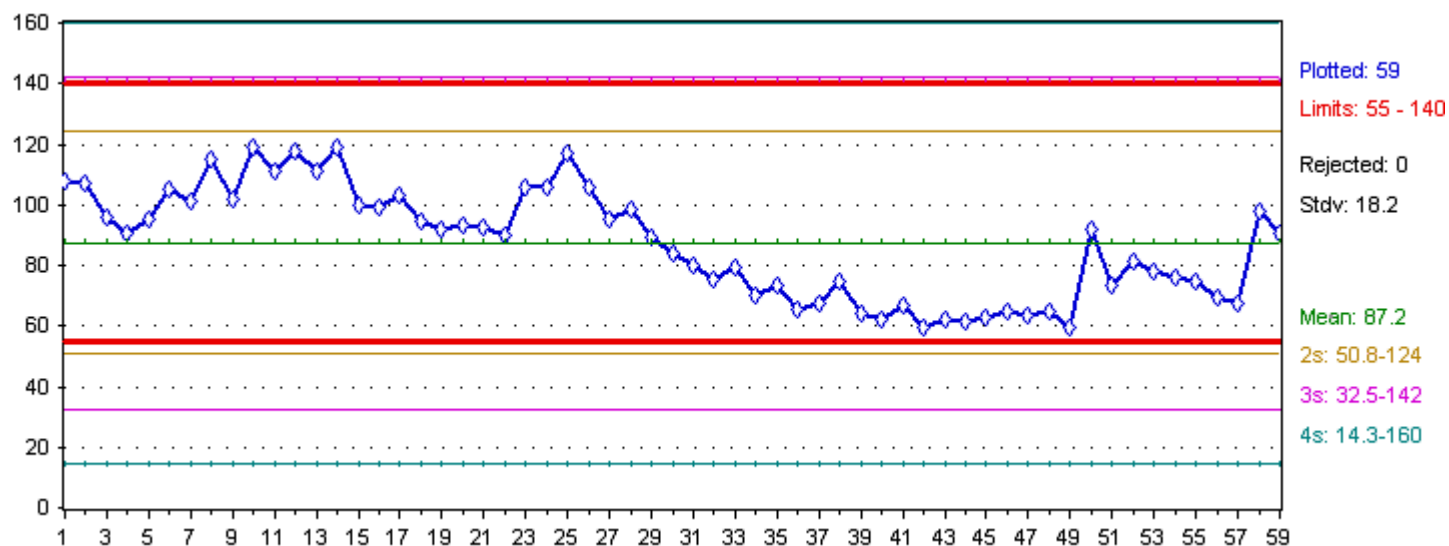
Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

Naphthalene

LCS %R



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3D15001-BS1	4/15/13	4/15/13	50	54.2	108.34	55-140	
	3D16001-BS1	4/16/13	4/16/13	50	53.6	107.24	55-140	
	3D17005-BS1	4/17/13	4/17/13	50	48	96.02	55-140	
	3D18016-BS1	4/18/13	4/18/13	50	45.2	90.44	55-140	
	3D19006-BS1	4/19/13	4/19/13	50	47.6	95.18	55-140	
	3D22004-BS1	4/22/13	4/22/13	50	52.3	104.58	55-140	
	3D23008-BS1	4/23/13	4/23/13	50	50.5	101.02	55-140	
	3D24009-BS1	4/24/13	4/24/13	50	57.6	115.24	55-140	
	3D25008-BS1	4/25/13	4/25/13	50	50.9	101.76	55-140	
	3D26005-BS1	4/26/13	4/26/13	50	59.7	119.34	55-140	
	3D29008-BS1	4/29/13	4/29/13	50	55.7	111.46	55-140	
	3D30017-BS1	4/30/13	4/30/13	50	58.9	117.78	55-140	
	3D30025-BS1	4/30/13	4/30/13	50	55.6	111.2	55-140	
	3E01005-BS1	5/1/13	5/1/13	50	59.5	119.06	55-140	
	3E06002-BS1	5/6/13	5/6/13	50	49.9	99.86	55-140	
	3E07006-BS1	5/7/13	5/7/13	50	49.5	98.96	55-140	
	3E08004-BS1	5/8/13	5/8/13	50	51.6	103.14	55-140	
	3E08018-BS1	5/8/13	5/8/13	50	47.2	94.36	55-140	
	3E09011-BS1	5/9/13	5/9/13	50	46	92.08	55-140	
	3E10010-BS1	5/10/13	5/10/13	50	46.8	93.5	55-140	
	3E11006-BS1	5/11/13	5/11/13	50	46.4	92.84	55-140	
	3E13317-BS1	5/13/13	5/13/13	50	45	90.1	55-140	
	3E14013-BS1	5/14/13	5/14/13	50	53	105.92	55-140	
	3E14018-BS1	5/14/13	5/14/13	50	53	106.04	55-140	
	3E15010-BS1	5/15/13	5/15/13	50	58.4	116.82	55-140	
	3E16019-BS1	5/16/13	5/16/13	50	52.8	105.5	55-140	
	3E17010-BS1	5/17/13	5/17/13	50	47.7	95.44	55-140	

Printed: 07/13/2013 08:16

Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

<i>Naphthalene</i>		<i>LCS %R</i>					Limits	Qualifiers
Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R		
	3E17016-BS1	5/17/13	5/17/13	50	49.4	98.8	55-140	
	3E18005-BS1	5/18/13	5/18/13	50	44.8	89.54	55-140	
	3E20006-BS1	5/20/13	5/20/13	50	41.8	83.68	55-140	
	3E20018-BS1	5/20/13	5/20/13	50	40.1	80.18	55-140	
	3E21009-BS1	5/21/13	5/21/13	50	37.8	75.6	55-140	
	3E21026-BS1	5/21/13	5/21/13	50	39.6	79.16	55-140	
	3E22016-BS1	5/22/13	5/22/13	50	35	70.06	55-140	
	3E22027-BS1	5/22/13	5/22/13	50	36.8	73.7	55-140	
	3E23011-BS1	5/23/13	5/23/13	50	32.8	65.64	55-140	
	3E23019-BS1	5/23/13	5/23/13	50	33.8	67.58	55-140	
	3E24013-BS1	5/24/13	5/24/13	50	37.3	74.68	55-140	
	3E24019-BS1	5/24/13	5/24/13	50	32	63.96	55-140	
	3E25004-BS1	5/25/13	5/25/13	50	31.2	62.38	55-140	
	3E25008-BS1	5/26/13	5/26/13	50	33.5	67	55-140	
	3E28013-BS1	5/28/13	5/28/13	50	29.7	59.42	55-140	
	3E28023-BS1	5/28/13	5/28/13	50	31.2	62.42	55-140	
	3E29016-BS1	5/29/13	5/29/13	50	30.7	61.4	55-140	
	3E30015-BS1	5/30/13	5/30/13	50	31.3	62.54	55-140	
	3E31003-BS1	5/31/13	5/31/13	50	32.5	64.92	55-140	
	3E31016-BS1	5/31/13	5/31/13	50	31.7	63.38	55-140	
	3F01002-BS1	6/1/13	6/1/13	50	32.3	64.52	55-140	
	3F01003-BS1	6/1/13	6/1/13	50	29.7	59.42	55-140	
	3F03022-BS1	6/3/13	6/3/13	50	45.8	91.6	55-140	
	3F04012-BS1	6/4/13	6/4/13	50	36.8	73.64	55-140	
	3F04013-BS1	6/4/13	6/4/13	50	40.6	81.24	55-140	
	3F05001-BS1	6/5/13	6/5/13	50	39.1	78.14	55-140	
	3F05018-BS1	6/5/13	6/5/13	50	38	76.02	55-140	
	3F06010-BS1	6/6/13	6/6/13	50	37.4	74.76	55-140	
	3F07001-BS1	6/7/13	6/7/13	50	34.6	69.24	55-140	
	3F07034-BS1	6/7/13	6/7/13	50	33.7	67.36	55-140	
	3F13017-BS1	6/13/13	6/13/13	50	49	98.08	55-140	
	3F13024-BS1	6/13/13	6/13/13	50	45.1	90.26	55-140	

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Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

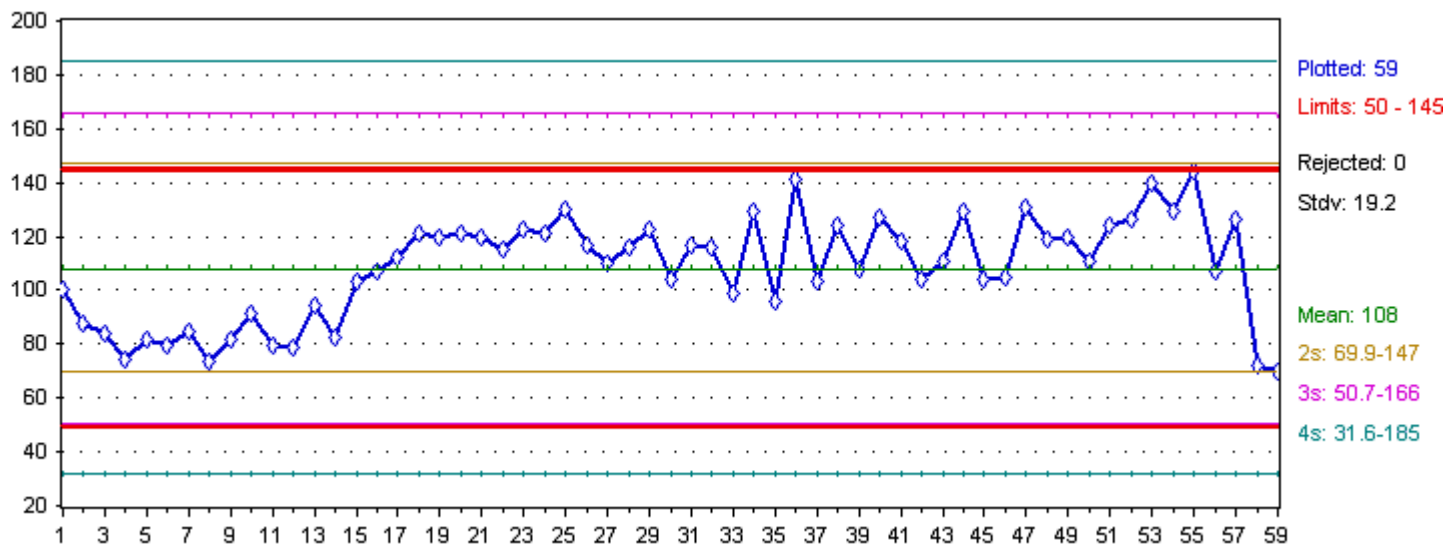
Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

Vinyl chloride

LCS %R



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3D15001-BS1	4/15/13	4/15/13	50	50.1	100.26	50-145	
	3D16001-BS1	4/16/13	4/16/13	50	43.9	87.82	50-145	
	3D17005-BS1	4/17/13	4/17/13	50	42	84.02	50-145	
	3D18016-BS1	4/18/13	4/18/13	50	37.1	74.16	50-145	
	3D19006-BS1	4/19/13	4/19/13	50	40.7	81.48	50-145	
	3D22004-BS1	4/22/13	4/22/13	50	39.6	79.2	50-145	
	3D23008-BS1	4/23/13	4/23/13	50	42.4	84.78	50-145	
	3D24009-BS1	4/24/13	4/24/13	50	36.7	73.32	50-145	
	3D25008-BS1	4/25/13	4/25/13	50	40.7	81.36	50-145	
	3D26005-BS1	4/26/13	4/26/13	50	45.6	91.2	50-145	
	3D29008-BS1	4/29/13	4/29/13	50	39.9	79.84	50-145	
	3D30017-BS1	4/30/13	4/30/13	50	39.6	79.12	50-145	
	3D30025-BS1	4/30/13	4/30/13	50	47.3	94.62	50-145	
	3E01005-BS1	5/1/13	5/1/13	50	41.2	82.44	50-145	
	3E06002-BS1	5/6/13	5/6/13	50	51.5	103	50-145	
	3E07006-BS1	5/7/13	5/7/13	50	53.3	106.6	50-145	
	3E08004-BS1	5/8/13	5/8/13	50	56.1	112.2	50-145	
	3E08018-BS1	5/8/13	5/8/13	50	60.4	120.84	50-145	
	3E09011-BS1	5/9/13	5/9/13	50	60.1	120.22	50-145	
	3E10010-BS1	5/10/13	5/10/13	50	60.6	121.14	50-145	
	3E11006-BS1	5/11/13	5/11/13	50	60.2	120.4	50-145	
	3E13317-BS1	5/13/13	5/13/13	50	57.6	115.14	50-145	
	3E14013-BS1	5/14/13	5/14/13	50	61.6	123.26	50-145	
	3E14018-BS1	5/14/13	5/14/13	50	60.7	121.48	50-145	
	3E15010-BS1	5/15/13	5/15/13	50	65	129.9	50-145	
	3E16019-BS1	5/16/13	5/16/13	50	58.6	117.16	50-145	
	3E17010-BS1	5/17/13	5/17/13	50	54.9	109.8	50-145	

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Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

<i>Vinyl chloride</i>		<i>LCS %R</i>					Limits	Qualifiers
Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R		
	3E17016-BS1	5/17/13	5/17/13	50	58.2	116.3	50-145	
	3E18005-BS1	5/18/13	5/18/13	50	61.4	122.8	50-145	
	3E20006-BS1	5/20/13	5/20/13	50	51.8	103.52	50-145	
	3E20018-BS1	5/20/13	5/20/13	50	58.7	117.48	50-145	
	3E21009-BS1	5/21/13	5/21/13	50	57.9	115.76	50-145	
	3E21026-BS1	5/21/13	5/21/13	50	49.5	99	50-145	
	3E22016-BS1	5/22/13	5/22/13	50	64.5	128.98	50-145	
	3E22027-BS1	5/22/13	5/22/13	50	47.8	95.66	50-145	
	3E23011-BS1	5/23/13	5/23/13	50	70.4	140.86	50-145	
	3E23019-BS1	5/23/13	5/23/13	50	51.3	102.6	50-145	
	3E24013-BS1	5/24/13	5/24/13	50	62	123.94	50-145	
	3E24019-BS1	5/24/13	5/24/13	50	54.1	108.12	50-145	
	3E25004-BS1	5/25/13	5/25/13	50	63.7	127.4	50-145	
	3E25008-BS1	5/26/13	5/26/13	50	59.1	118.12	50-145	
	3E28013-BS1	5/28/13	5/28/13	50	51.8	103.5	50-145	
	3E28023-BS1	5/28/13	5/28/13	50	55.3	110.52	50-145	
	3E29016-BS1	5/29/13	5/29/13	50	64.3	128.56	50-145	
	3E30015-BS1	5/30/13	5/30/13	50	52.1	104.14	50-145	
	3E31003-BS1	5/31/13	5/31/13	50	52.4	104.74	50-145	
	3E31016-BS1	5/31/13	5/31/13	50	65.5	130.96	50-145	
	3F01002-BS1	6/1/13	6/1/13	50	59.7	119.48	50-145	
	3F01003-BS1	6/1/13	6/1/13	50	60.2	120.36	50-145	
	3F03022-BS1	6/3/13	6/3/13	50	55.7	111.42	50-145	
	3F04012-BS1	6/4/13	6/4/13	50	61.8	123.56	50-145	
	3F04013-BS1	6/4/13	6/4/13	50	63	126.06	50-145	
	3F05001-BS1	6/5/13	6/5/13	50	69.8	139.52	50-145	
	3F05018-BS1	6/5/13	6/5/13	50	64.6	129.3	50-145	
	3F06010-BS1	6/6/13	6/6/13	50	72.2	144.46	50-145	
	3F07001-BS1	6/7/13	6/7/13	50	53.5	107.02	50-145	
	3F07034-BS1	6/7/13	6/7/13	50	62.8	125.68	50-145	
	3F13017-BS1	6/13/13	6/13/13	50	36.1	72.18	50-145	
	3F13024-BS1	6/13/13	6/13/13	50	34.8	69.66	50-145	

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Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

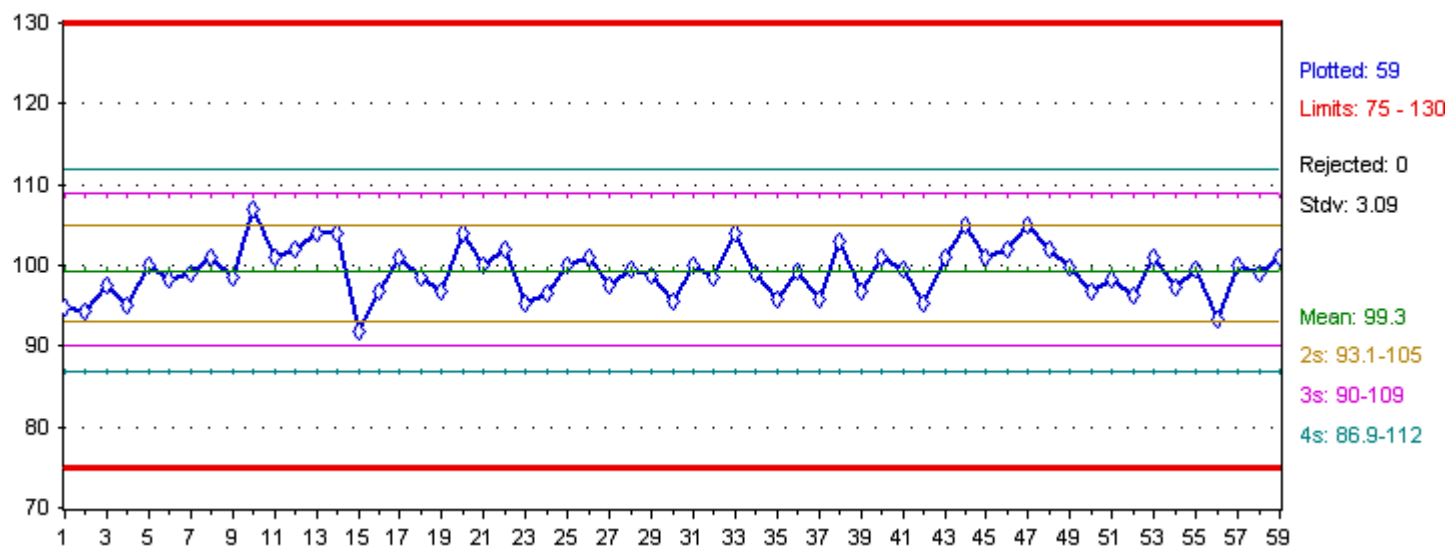
Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

Xylenes (total)

LCS %R



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3D15001-BS1	4/15/13	4/15/13	150	142	94.76	75-130	
	3D16001-BS1	4/16/13	4/16/13	150	141	94.24	75-130	
	3D17005-BS1	4/17/13	4/17/13	150	146	97.51334	75-130	
	3D18016-BS1	4/18/13	4/18/13	150	143	95.12	75-130	
	3D19006-BS1	4/19/13	4/19/13	150	150	99.86667	75-130	
	3D22004-BS1	4/22/13	4/22/13	150	147	98.32	75-130	
	3D23008-BS1	4/23/13	4/23/13	150	149	99.12667	75-130	
	3D24009-BS1	4/24/13	4/24/13	150	151	100.6867	75-130	
	3D25008-BS1	4/25/13	4/25/13	150	148	98.35333	75-130	
	3D26005-BS1	4/26/13	4/26/13	150	161	107.2267	75-130	
	3D29008-BS1	4/29/13	4/29/13	150	152	101.0867	75-130	
	3D30017-BS1	4/30/13	4/30/13	150	153	101.9467	75-130	
	3D30025-BS1	4/30/13	4/30/13	150	156	103.9867	75-130	
	3E01005-BS1	5/1/13	5/1/13	150	156	103.98	75-130	
	3E06002-BS1	5/6/13	5/6/13	150	138	91.94667	75-130	
	3E07006-BS1	5/7/13	5/7/13	150	145	96.68667	75-130	
	3E08004-BS1	5/8/13	5/8/13	150	152	101	75-130	
	3E08018-BS1	5/8/13	5/8/13	150	148	98.58667	75-130	
	3E09011-BS1	5/9/13	5/9/13	150	145	96.7	75-130	
	3E10010-BS1	5/10/13	5/10/13	150	156	103.8067	75-130	
	3E11006-BS1	5/11/13	5/11/13	150	150	100.3067	75-130	
	3E13317-BS1	5/13/13	5/13/13	150	152	101.5	75-130	
	3E14013-BS1	5/14/13	5/14/13	150	143	95.41333	75-130	
	3E14018-BS1	5/14/13	5/14/13	150	145	96.56667	75-130	
	3E15010-BS1	5/15/13	5/15/13	150	150	100.24	75-130	
	3E16019-BS1	5/16/13	5/16/13	150	152	101.0667	75-130	
	3E17010-BS1	5/17/13	5/17/13	150	146	97.62	75-130	

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Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

<i>Xylenes (total)</i>			<i>LCS %R</i>				Limits	Qualifiers
Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R		
	3E17016-BS1	5/17/13	5/17/13	150	149	99.64667	75-130	
	3E18005-BS1	5/18/13	5/18/13	150	148	98.8	75-130	
	3E20006-BS1	5/20/13	5/20/13	150	143	95.48	75-130	
	3E20018-BS1	5/20/13	5/20/13	150	150	99.87333	75-130	
	3E21009-BS1	5/21/13	5/21/13	150	148	98.50666	75-130	
	3E21026-BS1	5/21/13	5/21/13	150	156	104.1	75-130	
	3E22016-BS1	5/22/13	5/22/13	150	148	98.97334	75-130	
	3E22027-BS1	5/22/13	5/22/13	150	144	95.68	75-130	
	3E23011-BS1	5/23/13	5/23/13	150	149	99.2	75-130	
	3E23019-BS1	5/23/13	5/23/13	150	144	95.90667	75-130	
	3E24013-BS1	5/24/13	5/24/13	150	154	102.96	75-130	
	3E24019-BS1	5/24/13	5/24/13	150	145	96.80667	75-130	
	3E25004-BS1	5/25/13	5/25/13	150	151	100.7933	75-130	
	3E25008-BS1	5/26/13	5/26/13	150	149	99.48	75-130	
	3E28013-BS1	5/28/13	5/28/13	150	143	95.16	75-130	
	3E28023-BS1	5/28/13	5/28/13	150	151	100.9867	75-130	
	3E29016-BS1	5/29/13	5/29/13	150	157	104.8867	75-130	
	3E30015-BS1	5/30/13	5/30/13	150	151	100.8867	75-130	
	3E31003-BS1	5/31/13	5/31/13	150	152	101.6067	75-130	
	3E31016-BS1	5/31/13	5/31/13	150	157	104.8533	75-130	
	3F01002-BS1	6/1/13	6/1/13	150	153	101.88	75-130	
	3F01003-BS1	6/1/13	6/1/13	150	150	99.75334	75-130	
	3F03022-BS1	6/3/13	6/3/13	150	145	96.82	75-130	
	3F04012-BS1	6/4/13	6/4/13	150	147	98.24666	75-130	
	3F04013-BS1	6/4/13	6/4/13	150	145	96.40667	75-130	
	3F05001-BS1	6/5/13	6/5/13	150	151	100.6333	75-130	
	3F05018-BS1	6/5/13	6/5/13	150	146	97.30667	75-130	
	3F06010-BS1	6/6/13	6/6/13	150	149	99.50666	75-130	
	3F07001-BS1	6/7/13	6/7/13	150	140	93.44	75-130	
	3F07034-BS1	6/7/13	6/7/13	150	150	100.32	75-130	
	3F13017-BS1	6/13/13	6/13/13	150	148	98.98666	75-130	
	3F13024-BS1	6/13/13	6/13/13	150	151	100.62	75-130	

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Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

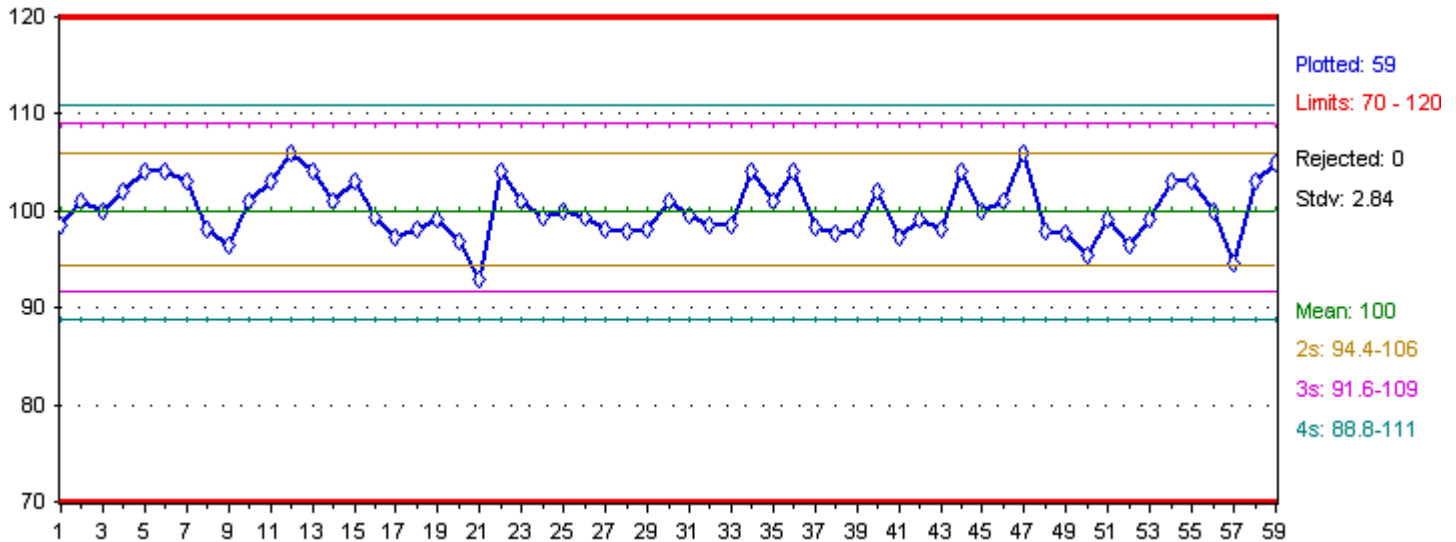
Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

1,2-Dichloroethane-d4

%R in BLK



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3D15001-BLK1	4/15/13	4/15/13	30	29.55	98.5	70-120	
	3D16001-BLK1	4/16/13	4/16/13	30	30.37	101.2333	70-120	
	3D17005-BLK1	4/17/13	4/17/13	30	30.13	100.4333	70-120	
	3D18016-BLK1	4/18/13	4/18/13	30	30.72	102.4	70-120	
	3D19006-BLK1	4/19/13	4/19/13	30	31.1	103.6667	70-120	
	3D22004-BLK1	4/22/13	4/22/13	30	31.18	103.9333	70-120	
	3D23008-BLK1	4/23/13	4/23/13	30	31	103.3333	70-120	
	3D24009-BLK1	4/24/13	4/24/13	30	29.43	98.1	70-120	
	3D25008-BLK1	4/25/13	4/25/13	30	28.95	96.5	70-120	
	3D26005-BLK1	4/26/13	4/26/13	30	30.25	100.8333	70-120	
	3D29008-BLK1	4/29/13	4/29/13	30	30.86	102.8667	70-120	
	3D30017-BLK1	4/30/13	4/30/13	30	31.66	105.5333	70-120	
	3D30025-BLK1	4/30/13	4/30/13	30	31.16	103.8667	70-120	
	3E01005-BLK1	5/1/13	5/1/13	30	30.24	100.8	70-120	
	3E06002-BLK1	5/6/13	5/6/13	30	30.82	102.7333	70-120	
	3E07006-BLK1	5/7/13	5/7/13	30	29.79	99.3	70-120	
	3E08004-BLK1	5/8/13	5/8/13	30	29.18	97.26667	70-120	
	3E08018-BLK1	5/8/13	5/8/13	30	29.47	98.23333	70-120	
	3E09011-BLK1	5/9/13	5/9/13	30	29.76	99.2	70-120	
	3E10010-BLK1	5/10/13	5/10/13	30	29.04	96.8	70-120	
	3E11006-BLK1	5/11/13	5/11/13	30	27.91	93.03333	70-120	
	3E13317-BLK1	5/13/13	5/13/13	30	31.09	103.6333	70-120	
	3E14013-BLK1	5/14/13	5/14/13	30	30.17	100.5667	70-120	
	3E14018-BLK1	5/14/13	5/14/13	30	29.81	99.36666	70-120	
	3E15010-BLK1	5/15/13	5/15/13	30	30.01	100.0333	70-120	
	3E16019-BLK1	5/16/13	5/16/13	30	29.79	99.3	70-120	
	3E17010-BLK1	5/17/13	5/17/13	30	29.46	98.2	70-120	
	3E17016-BLK1	5/18/13	5/18/13	30	29.33	97.76667	70-120	
	3E18005-BLK1	5/18/13	5/18/13	30	29.46	98.2	70-120	

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Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

1,2-Dichloroethane-d4

%R in BLK

Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3E20006-BLK1	5/20/13	5/20/13	30	30.16	100.5333	70-120	
	3E20018-BLK1	5/20/13	5/20/13	30	29.89	99.63333	70-120	
	3E21009-BLK1	5/21/13	5/21/13	30	29.57	98.56667	70-120	
	3E21026-BLK1	5/21/13	5/21/13	30	29.55	98.5	70-120	
	3E22016-BLK1	5/22/13	5/22/13	30	31.31	104.3667	70-120	
	3E22027-BLK1	5/22/13	5/22/13	30	30.24	100.8	70-120	
	3E23011-BLK1	5/23/13	5/23/13	30	31.15	103.8333	70-120	
	3E23019-BLK1	5/23/13	5/23/13	30	29.51	98.36667	70-120	
	3E24013-BLK1	5/24/13	5/24/13	30	29.28	97.6	70-120	
	3E24019-BLK1	5/25/13	5/25/13	30	29.41	98.03333	70-120	
	3E25004-BLK1	5/25/13	5/25/13	30	30.58	101.9333	70-120	
	3E25008-BLK1	5/26/13	5/26/13	30	29.2	97.33334	70-120	
	3E28013-BLK1	5/28/13	5/28/13	30	29.77	99.23333	70-120	
	3E28023-BLK1	5/28/13	5/28/13	30	29.39	97.96666	70-120	
	3E29016-BLK1	5/29/13	5/29/13	30	31.09	103.6333	70-120	
	3E30015-BLK1	5/30/13	5/30/13	30	30.02	100.0667	70-120	
	3E31003-BLK1	5/31/13	5/31/13	30	30.2	100.6667	70-120	
	3E31016-BLK1	5/31/13	5/31/13	30	31.77	105.9	70-120	
	3F01002-BLK1	6/1/13	6/1/13	30	29.33	97.76667	70-120	
	3F01003-BLK1	6/2/13	6/2/13	30	29.27	97.56667	70-120	
	3F03022-BLK1	6/3/13	6/3/13	30	28.63	95.43333	70-120	
	3F04012-BLK1	6/4/13	6/4/13	30	29.76	99.2	70-120	
	3F04013-BLK1	6/4/13	6/4/13	30	28.96	96.53333	70-120	
	3F05001-BLK1	6/5/13	6/5/13	30	29.75	99.16667	70-120	
	3F05018-BLK1	6/5/13	6/5/13	30	30.92	103.0667	70-120	
	3F06010-BLK1	6/6/13	6/6/13	30	31.03	103.4333	70-120	
	3F07001-BLK1	6/7/13	6/7/13	30	29.98	99.93333	70-120	
	3F07034-BLK1	6/7/13	6/7/13	30	28.36	94.53334	70-120	
	3F13017-BLK1	6/13/13	6/13/13	30	30.94	103.1333	70-120	
	3F13024-BLK1	6/14/13	6/14/13	30	31.46	104.8667	70-120	

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Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

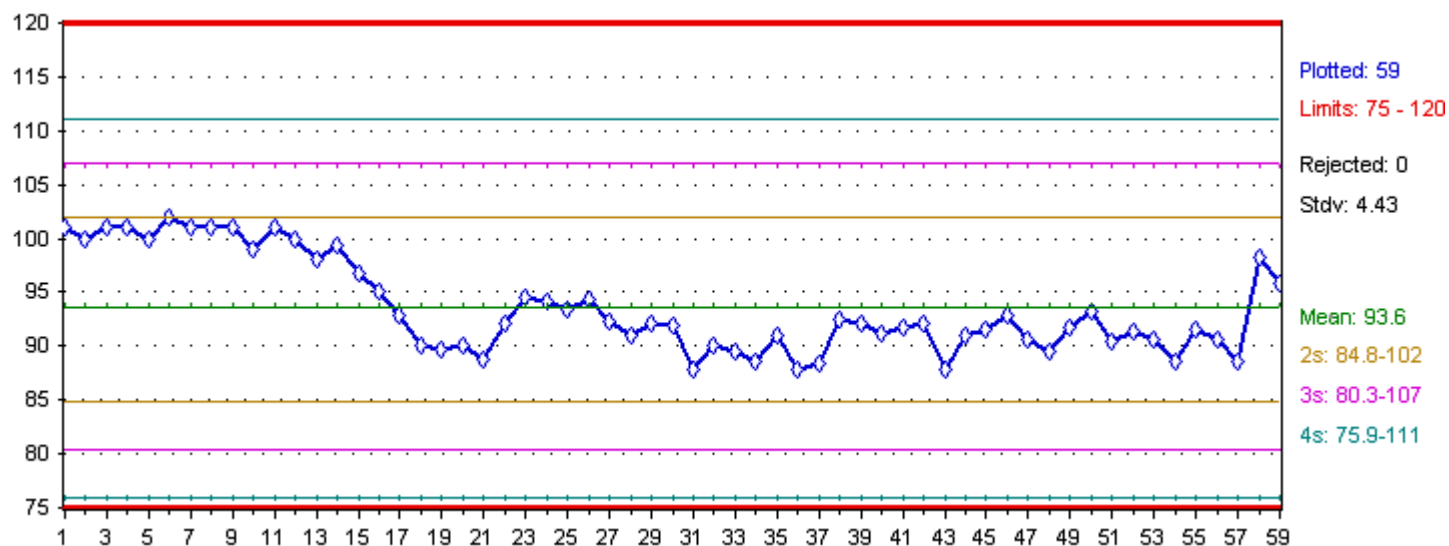
Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

Bromofluorobenzene

%R in BLK



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3D15001-BLK1	4/15/13	4/15/13	30	30.43	101.4333	75-120	
	3D16001-BLK1	4/16/13	4/16/13	30	30.05	100.1667	75-120	
	3D17005-BLK1	4/17/13	4/17/13	30	30.24	100.8	75-120	
	3D18016-BLK1	4/18/13	4/18/13	30	30.42	101.4	75-120	
	3D19006-BLK1	4/19/13	4/19/13	30	30.13	100.4333	75-120	
	3D22004-BLK1	4/22/13	4/22/13	30	30.52	101.7333	75-120	
	3D23008-BLK1	4/23/13	4/23/13	30	30.43	101.4333	75-120	
	3D24009-BLK1	4/24/13	4/24/13	30	30.37	101.2333	75-120	
	3D25008-BLK1	4/25/13	4/25/13	30	30.27	100.9	75-120	
	3D26005-BLK1	4/26/13	4/26/13	30	29.67	98.9	75-120	
	3D29008-BLK1	4/29/13	4/29/13	30	30.33	101.1	75-120	
	3D30017-BLK1	4/30/13	4/30/13	30	30.07	100.2333	75-120	
	3D30025-BLK1	4/30/13	4/30/13	30	29.44	98.13334	75-120	
	3E01005-BLK1	5/1/13	5/1/13	30	29.83	99.43333	75-120	
	3E06002-BLK1	5/6/13	5/6/13	30	29.02	96.73333	75-120	
	3E07006-BLK1	5/7/13	5/7/13	30	28.52	95.06667	75-120	
	3E08004-BLK1	5/8/13	5/8/13	30	27.87	92.9	75-120	
	3E08018-BLK1	5/8/13	5/8/13	30	27.01	90.03333	75-120	
	3E09011-BLK1	5/9/13	5/9/13	30	26.9	89.66667	75-120	
	3E10010-BLK1	5/10/13	5/10/13	30	27.02	90.06667	75-120	
	3E11006-BLK1	5/11/13	5/11/13	30	26.64	88.8	75-120	
	3E13317-BLK1	5/13/13	5/13/13	30	27.66	92.2	75-120	
	3E14013-BLK1	5/14/13	5/14/13	30	28.39	94.63333	75-120	
	3E14018-BLK1	5/14/13	5/14/13	30	28.25	94.16667	75-120	
	3E15010-BLK1	5/15/13	5/15/13	30	28.02	93.4	75-120	
	3E16019-BLK1	5/16/13	5/16/13	30	28.31	94.36666	75-120	
	3E17010-BLK1	5/17/13	5/17/13	30	27.68	92.26667	75-120	
	3E17016-BLK1	5/18/13	5/18/13	30	27.3	91	75-120	
	3E18005-BLK1	5/18/13	5/18/13	30	27.63	92.1	75-120	

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Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

Bromofluorobenzene				%R in BLK			Limits	Qualifiers
Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R		
	3E20006-BLK1	5/20/13	5/20/13	30	27.58	91.93333	75-120	
	3E20018-BLK1	5/20/13	5/20/13	30	26.33	87.76667	75-120	
	3E21009-BLK1	5/21/13	5/21/13	30	26.99	89.96667	75-120	
	3E21026-BLK1	5/21/13	5/21/13	30	26.86	89.53334	75-120	
	3E22016-BLK1	5/22/13	5/22/13	30	26.57	88.56667	75-120	
	3E22027-BLK1	5/22/13	5/22/13	30	27.27	90.9	75-120	
	3E23011-BLK1	5/23/13	5/23/13	30	26.37	87.9	75-120	
	3E23019-BLK1	5/23/13	5/23/13	30	26.53	88.43334	75-120	
	3E24013-BLK1	5/24/13	5/24/13	30	27.73	92.43333	75-120	
	3E24019-BLK1	5/25/13	5/25/13	30	27.64	92.13333	75-120	
	3E25004-BLK1	5/25/13	5/25/13	30	27.33	91.1	75-120	
	3E25008-BLK1	5/26/13	5/26/13	30	27.52	91.73333	75-120	
	3E28013-BLK1	5/28/13	5/28/13	30	27.67	92.23333	75-120	
	3E28023-BLK1	5/28/13	5/28/13	30	26.38	87.93333	75-120	
	3E29016-BLK1	5/29/13	5/29/13	30	27.26	90.86667	75-120	
	3E30015-BLK1	5/30/13	5/30/13	30	27.48	91.6	75-120	
	3E31003-BLK1	5/31/13	5/31/13	30	27.84	92.8	75-120	
	3E31016-BLK1	5/31/13	5/31/13	30	27.21	90.7	75-120	
	3F01002-BLK1	6/1/13	6/1/13	30	26.84	89.46667	75-120	
	3F01003-BLK1	6/2/13	6/2/13	30	27.51	91.7	75-120	
	3F03022-BLK1	6/3/13	6/3/13	30	28	93.33333	75-120	
	3F04012-BLK1	6/4/13	6/4/13	30	27.15	90.5	75-120	
	3F04013-BLK1	6/4/13	6/4/13	30	27.38	91.26666	75-120	
	3F05001-BLK1	6/5/13	6/5/13	30	27.18	90.6	75-120	
	3F05018-BLK1	6/5/13	6/5/13	30	26.55	88.5	75-120	
	3F06010-BLK1	6/6/13	6/6/13	30	27.44	91.46667	75-120	
	3F07001-BLK1	6/7/13	6/7/13	30	27.19	90.63334	75-120	
	3F07034-BLK1	6/7/13	6/7/13	30	26.56	88.53333	75-120	
	3F13017-BLK1	6/13/13	6/13/13	30	29.46	98.2	75-120	
	3F13024-BLK1	6/14/13	6/14/13	30	28.75	95.83333	75-120	

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Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

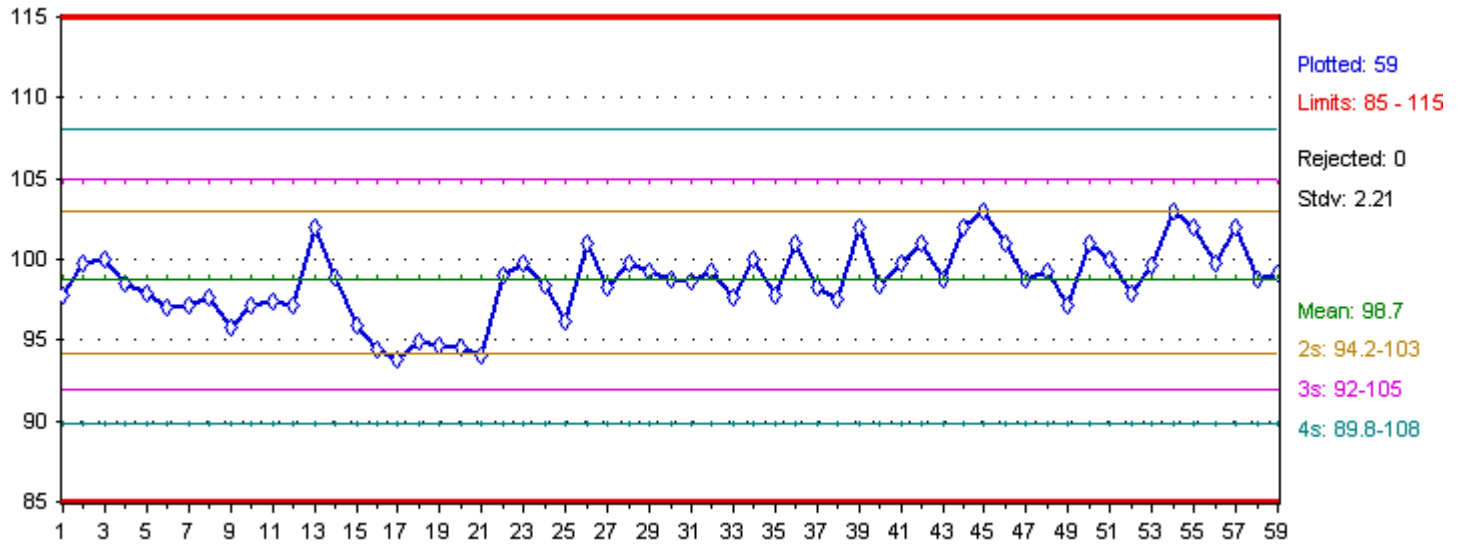
Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

Dibromofluoromethane

%R in BLK



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3D15001-BLK1	4/15/13	4/15/13	30	29.35	97.83333	85-115	
	3D16001-BLK1	4/16/13	4/16/13	30	29.95	99.83334	85-115	
	3D17005-BLK1	4/17/13	4/17/13	30	30.01	100.0333	85-115	
	3D18016-BLK1	4/18/13	4/18/13	30	29.56	98.53333	85-115	
	3D19006-BLK1	4/19/13	4/19/13	30	29.36	97.86667	85-115	
	3D22004-BLK1	4/22/13	4/22/13	30	29.09	96.96667	85-115	
	3D23008-BLK1	4/23/13	4/23/13	30	29.13	97.1	85-115	
	3D24009-BLK1	4/24/13	4/24/13	30	29.28	97.6	85-115	
	3D25008-BLK1	4/25/13	4/25/13	30	28.75	95.83333	85-115	
	3D26005-BLK1	4/26/13	4/26/13	30	29.17	97.23333	85-115	
	3D29008-BLK1	4/29/13	4/29/13	30	29.22	97.4	85-115	
	3D30017-BLK1	4/30/13	4/30/13	30	29.16	97.2	85-115	
	3D30025-BLK1	4/30/13	4/30/13	30	30.55	101.8333	85-115	
	3E01005-BLK1	5/1/13	5/1/13	30	29.66	98.86667	85-115	
	3E06002-BLK1	5/6/13	5/6/13	30	28.77	95.9	85-115	
	3E07006-BLK1	5/7/13	5/7/13	30	28.31	94.36666	85-115	
	3E08004-BLK1	5/8/13	5/8/13	30	28.14	93.8	85-115	
	3E08018-BLK1	5/8/13	5/8/13	30	28.47	94.9	85-115	
	3E09011-BLK1	5/9/13	5/9/13	30	28.42	94.73333	85-115	
	3E10010-BLK1	5/10/13	5/10/13	30	28.36	94.53334	85-115	
	3E11006-BLK1	5/11/13	5/11/13	30	28.24	94.13333	85-115	
	3E13317-BLK1	5/13/13	5/13/13	30	29.7	99	85-115	
	3E14013-BLK1	5/14/13	5/14/13	30	29.9	99.66667	85-115	
	3E14018-BLK1	5/14/13	5/14/13	30	29.51	98.36667	85-115	
	3E15010-BLK1	5/15/13	5/15/13	30	28.86	96.2	85-115	
	3E16019-BLK1	5/16/13	5/16/13	30	30.26	100.8667	85-115	
	3E17010-BLK1	5/17/13	5/17/13	30	29.49	98.3	85-115	
	3E17016-BLK1	5/18/13	5/18/13	30	29.93	99.76667	85-115	
	3E18005-BLK1	5/18/13	5/18/13	30	29.77	99.23333	85-115	

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Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

<i>Dibromofluoromethane</i>				%R in BLK			Limits	Qualifiers
Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R		
	3E20006-BLK1	5/20/13	5/20/13	30	29.61	98.7	85-115	
	3E20018-BLK1	5/20/13	5/20/13	30	29.57	98.56667	85-115	
	3E21009-BLK1	5/21/13	5/21/13	30	29.75	99.16667	85-115	
	3E21026-BLK1	5/21/13	5/21/13	30	29.3	97.66666	85-115	
	3E22016-BLK1	5/22/13	5/22/13	30	30	100	85-115	
	3E22027-BLK1	5/22/13	5/22/13	30	29.33	97.76667	85-115	
	3E23011-BLK1	5/23/13	5/23/13	30	30.15	100.5	85-115	
	3E23019-BLK1	5/23/13	5/23/13	30	29.5	98.33333	85-115	
	3E24013-BLK1	5/24/13	5/24/13	30	29.26	97.53333	85-115	
	3E24019-BLK1	5/25/13	5/25/13	30	30.46	101.5333	85-115	
	3E25004-BLK1	5/25/13	5/25/13	30	29.52	98.4	85-115	
	3E25008-BLK1	5/26/13	5/26/13	30	29.92	99.73333	85-115	
	3E28013-BLK1	5/28/13	5/28/13	30	30.33	101.1	85-115	
	3E28023-BLK1	5/28/13	5/28/13	30	29.62	98.73334	85-115	
	3E29016-BLK1	5/29/13	5/29/13	30	30.57	101.9	85-115	
	3E30015-BLK1	5/30/13	5/30/13	30	30.95	103.1667	85-115	
	3E31003-BLK1	5/31/13	5/31/13	30	30.35	101.1667	85-115	
	3E31016-BLK1	5/31/13	5/31/13	30	29.62	98.73334	85-115	
	3F01002-BLK1	6/1/13	6/1/13	30	29.75	99.16667	85-115	
	3F01003-BLK1	6/2/13	6/2/13	30	29.13	97.1	85-115	
	3F03022-BLK1	6/3/13	6/3/13	30	30.15	100.5	85-115	
	3F04012-BLK1	6/4/13	6/4/13	30	30.14	100.4667	85-115	
	3F04013-BLK1	6/4/13	6/4/13	30	29.37	97.9	85-115	
	3F05001-BLK1	6/5/13	6/5/13	30	29.88	99.6	85-115	
	3F05018-BLK1	6/5/13	6/5/13	30	31.02	103.4	85-115	
	3F06010-BLK1	6/6/13	6/6/13	30	30.7	102.3333	85-115	
	3F07001-BLK1	6/7/13	6/7/13	30	29.94	99.8	85-115	
	3F07034-BLK1	6/7/13	6/7/13	30	30.52	101.7333	85-115	
	3F13017-BLK1	6/13/13	6/13/13	30	29.65	98.83333	85-115	
	3F13024-BLK1	6/14/13	6/14/13	30	29.73	99.1	85-115	

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Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

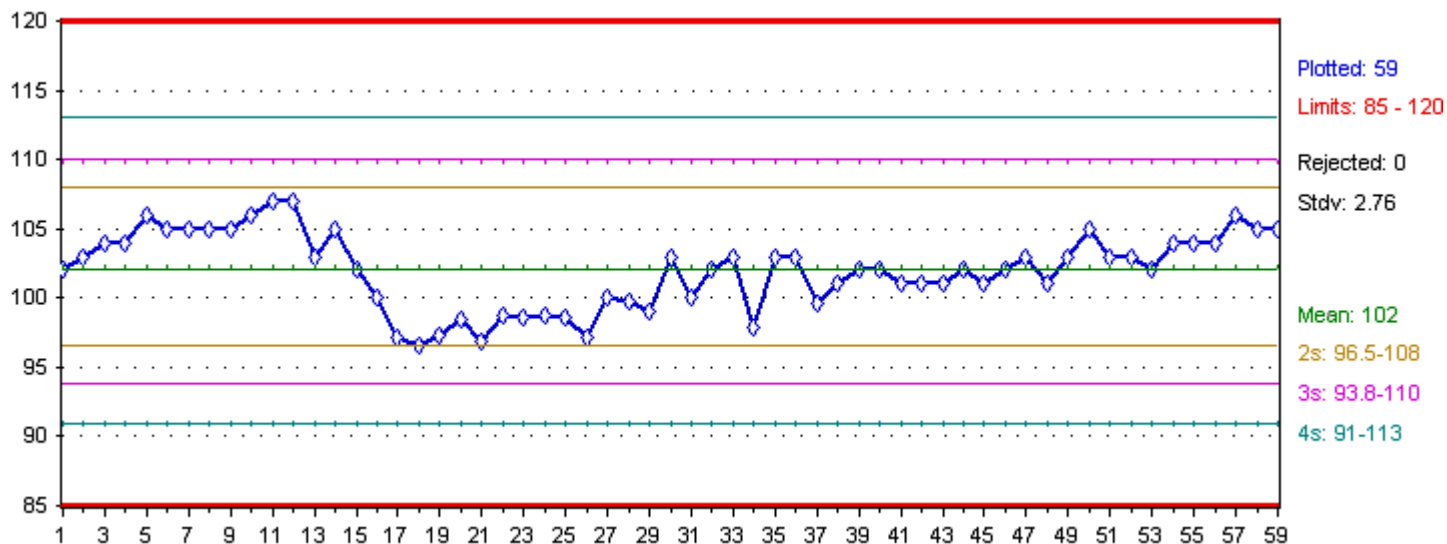
Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

Toluene-d8

%R in BLK



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3D15001-BLK1	4/15/13	4/15/13	30	30.66	102.2	85-120	
	3D16001-BLK1	4/16/13	4/16/13	30	30.97	103.2333	85-120	
	3D17005-BLK1	4/17/13	4/17/13	30	31.31	104.3667	85-120	
	3D18016-BLK1	4/18/13	4/18/13	30	31.09	103.6333	85-120	
	3D19006-BLK1	4/19/13	4/19/13	30	31.86	106.2	85-120	
	3D22004-BLK1	4/22/13	4/22/13	30	31.49	104.9667	85-120	
	3D23008-BLK1	4/23/13	4/23/13	30	31.57	105.2333	85-120	
	3D24009-BLK1	4/24/13	4/24/13	30	31.58	105.2667	85-120	
	3D25008-BLK1	4/25/13	4/25/13	30	31.6	105.3333	85-120	
	3D26005-BLK1	4/26/13	4/26/13	30	31.92	106.4	85-120	
	3D29008-BLK1	4/29/13	4/29/13	30	32.08	106.9333	85-120	
	3D30017-BLK1	4/30/13	4/30/13	30	32.14	107.1333	85-120	
	3D30025-BLK1	4/30/13	4/30/13	30	30.87	102.9	85-120	
	3E01005-BLK1	5/1/13	5/1/13	30	31.38	104.6	85-120	
	3E06002-BLK1	5/6/13	5/6/13	30	30.52	101.7333	85-120	
	3E07006-BLK1	5/7/13	5/7/13	30	30.03	100.1	85-120	
	3E08004-BLK1	5/8/13	5/8/13	30	29.17	97.23333	85-120	
	3E08018-BLK1	5/8/13	5/8/13	30	28.97	96.56666	85-120	
	3E09011-BLK1	5/9/13	5/9/13	30	29.2	97.33334	85-120	
	3E10010-BLK1	5/10/13	5/10/13	30	29.52	98.4	85-120	
	3E11006-BLK1	5/11/13	5/11/13	30	29.04	96.8	85-120	
	3E13317-BLK1	5/13/13	5/13/13	30	29.65	98.83333	85-120	
	3E14013-BLK1	5/14/13	5/14/13	30	29.59	98.63333	85-120	
	3E14018-BLK1	5/14/13	5/14/13	30	29.65	98.83333	85-120	
	3E15010-BLK1	5/15/13	5/15/13	30	29.58	98.6	85-120	
	3E16019-BLK1	5/16/13	5/16/13	30	29.17	97.23333	85-120	
	3E17010-BLK1	5/17/13	5/17/13	30	30	100	85-120	
	3E17016-BLK1	5/18/13	5/18/13	30	29.92	99.73333	85-120	
	3E18005-BLK1	5/18/13	5/18/13	30	29.73	99.1	85-120	

Printed: 07/13/2013 08:49

Matrices: Water

Client: All Clients

Instruments: gcmsvoa5

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: 5030B

Toluene-d8		%R in BLK					Limits	Qualifiers
Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R		
	3E20006-BLK1	5/20/13	5/20/13	30	30.8	102.6667	85-120	
	3E20018-BLK1	5/20/13	5/20/13	30	30.12	100.4	85-120	
	3E21009-BLK1	5/21/13	5/21/13	30	30.55	101.8333	85-120	
	3E21026-BLK1	5/21/13	5/21/13	30	30.77	102.5667	85-120	
	3E22016-BLK1	5/22/13	5/22/13	30	29.35	97.83333	85-120	
	3E22027-BLK1	5/22/13	5/22/13	30	30.94	103.1333	85-120	
	3E23011-BLK1	5/23/13	5/23/13	30	30.76	102.5333	85-120	
	3E23019-BLK1	5/23/13	5/23/13	30	29.87	99.56667	85-120	
	3E24013-BLK1	5/24/13	5/24/13	30	30.44	101.4667	85-120	
	3E24019-BLK1	5/25/13	5/25/13	30	30.66	102.2	85-120	
	3E25004-BLK1	5/25/13	5/25/13	30	30.45	101.5	85-120	
	3E25008-BLK1	5/26/13	5/26/13	30	30.29	100.9667	85-120	
	3E28013-BLK1	5/28/13	5/28/13	30	30.44	101.4667	85-120	
	3E28023-BLK1	5/28/13	5/28/13	30	30.34	101.1333	85-120	
	3E29016-BLK1	5/29/13	5/29/13	30	30.45	101.5	85-120	
	3E30015-BLK1	5/30/13	5/30/13	30	30.34	101.1333	85-120	
	3E31003-BLK1	5/31/13	5/31/13	30	30.54	101.8	85-120	
	3E31016-BLK1	5/31/13	5/31/13	30	30.84	102.8	85-120	
	3F01002-BLK1	6/1/13	6/1/13	30	30.26	100.8667	85-120	
	3F01003-BLK1	6/2/13	6/2/13	30	30.86	102.8667	85-120	
	3F03022-BLK1	6/3/13	6/3/13	30	31.49	104.9667	85-120	
	3F04012-BLK1	6/4/13	6/4/13	30	31.03	103.4333	85-120	
	3F04013-BLK1	6/4/13	6/4/13	30	30.92	103.0667	85-120	
	3F05001-BLK1	6/5/13	6/5/13	30	30.63	102.1	85-120	
	3F05018-BLK1	6/5/13	6/5/13	30	31.14	103.8	85-120	
	3F06010-BLK1	6/6/13	6/6/13	30	31.3	104.3333	85-120	
	3F07001-BLK1	6/7/13	6/7/13	30	31.23	104.1	85-120	
	3F07034-BLK1	6/7/13	6/7/13	30	31.89	106.3	85-120	
	3F13017-BLK1	6/13/13	6/13/13	30	31.52	105.0667	85-120	
	3F13024-BLK1	6/14/13	6/14/13	30	31.38	104.6	85-120	

Printed: 07/14/2013 07:20

Matrices: Water

Client: All Clients

Instruments: All Instruments

Project: All Projects

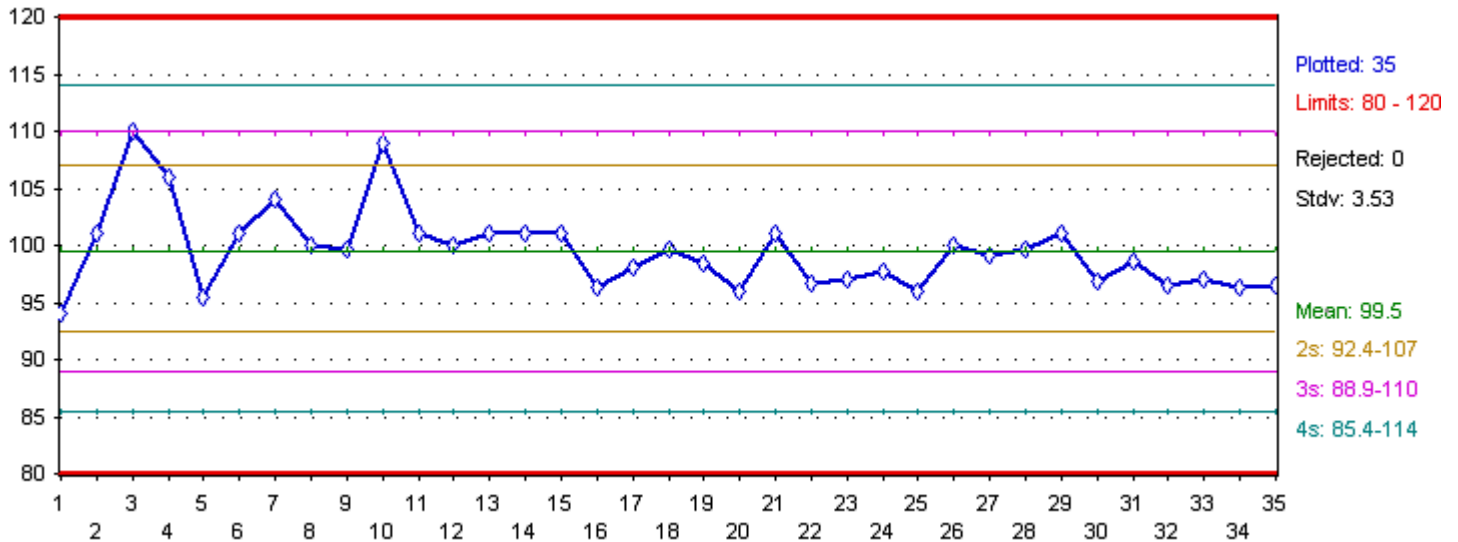
Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: All Extractions

Aluminum

LCS %R



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3A04011-BS1	1/4/13	1/8/13	2000	1882	94.095	80-120	
	3A16004-BS1	1/16/13	1/18/13	2000	2022	101.11	80-120	
	3A21015-BS1	1/21/13	1/23/13	2000	2192	109.615	80-120	
	3A22016-BS1	1/22/13	1/23/13	2000	2116	105.78	80-120	
	3A29015-BS1	1/29/13	1/30/13	2000	1910	95.5	80-120	
	3A29017-BS1	1/29/13	2/5/13	2000	2028	101.4	80-120	
	3A30005-BS1	1/30/13	2/5/13	2000	2087	104.33	80-120	
	3B05009-BS1	2/5/13	2/6/13	2000	2000	100.005	80-120	
	3B05010-BS1	2/5/13	2/6/13	2000	1993	99.665	80-120	
	3B20010-BS1	2/20/13	2/21/13	2000	2186	109.28	80-120	
	3B22012-BS1	2/22/13	2/26/13	2000	2010	100.5	80-120	
	3B26017-BS1	2/26/13	2/27/13	2000	2003	100.165	80-120	
	3B27018-BS1	2/27/13	2/28/13	2000	2013	100.64	80-120	
	3C01009-BS1	3/1/13	3/4/13	2000	2023	101.16	80-120	
	3C26009-BS1	3/26/13	3/27/13	2000	2026	101.315	80-120	
	3D15004-BS1	4/15/13	4/17/13	2000	1926	96.3	80-120	
	3D16009-BS1	4/16/13	4/17/13	2000	1962	98.095	80-120	
	3D19007-BS1	4/19/13	4/22/13	2000	1993	99.67	80-120	
	3D19008-BS1	4/19/13	4/22/13	2000	1969	98.47	80-120	
	3E14003-BS1	5/14/13	5/15/13	2000	1917	95.87	80-120	
	3E20004-BS1	5/20/13	5/21/13	2000	2015	100.77	80-120	
	3E20005-BS1	5/20/13	5/21/13	2000	1934	96.72	80-120	
	3E21005-BS1	5/21/13	5/22/13	2000	1941	97.07	80-120	
	3E21006-BS1	5/21/13	5/22/13	2000	1956	97.825	80-120	
	3E28011-BS1	5/28/13	5/31/13	2000	1918	95.905	80-120	
	3E29001-BS1	5/29/13	6/3/13	2000	2000	100.01	80-120	
	3F03025-BS1	6/3/13	6/10/13	2000	1984	99.195	80-120	

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Matrices: Water

Client: All Clients

Instruments: All Instruments

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: All Extractions

Aluminum

LCS %R

Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3F11001-BS1	6/11/13	6/12/13	2000	1993	99.655	80-120	
	3F11002-BS1	6/11/13	6/13/13	2000	2024	101.195	80-120	
	3F27001-BS1	6/27/13	7/1/13	2000	1935	96.765	80-120	
	3F27002-BS1	6/27/13	7/1/13	2000	1971	98.55	80-120	
	3F27003-BS1	6/27/13	6/28/13	2000	1931	96.535	80-120	
	3G02004-BS1	7/2/13	7/3/13	2000	1942	97.085	80-120	
	3G02005-BS1	7/2/13	7/3/13	2000	1925	96.265	80-120	
	3G02017-BS1	7/2/13	7/10/13	2000	1929	96.455	80-120	

Printed: 07/14/2013 07:20

Matrices: Water

Client: All Clients

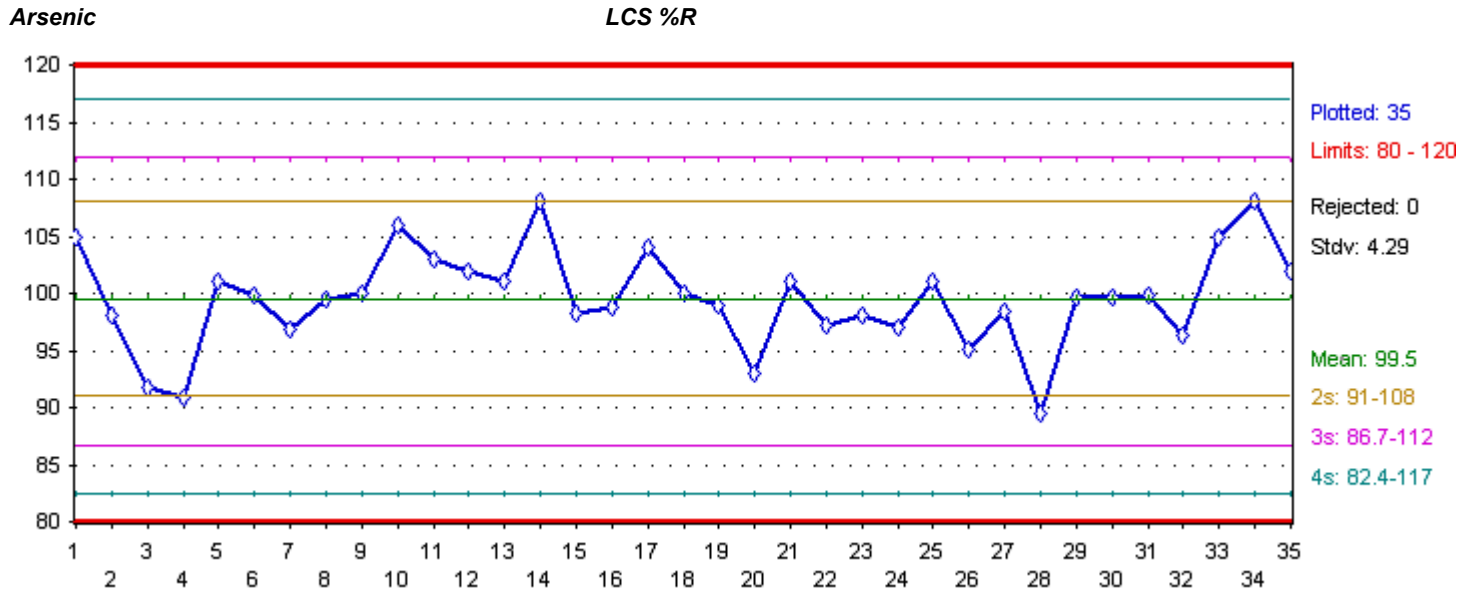
Instruments: All Instruments

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: All Extractions



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3A04011-BS1	1/4/13	1/8/13	250	263.7	105.48	80-120	
	3A16004-BS1	1/16/13	1/18/13	250	245.2	98.088	80-120	
	3A21015-BS1	1/21/13	1/23/13	250	229.6	91.848	80-120	
	3A22016-BS1	1/22/13	1/23/13	250	227.2	90.88	80-120	
	3A29015-BS1	1/29/13	1/30/13	250	253.6	101.452	80-120	
	3A29017-BS1	1/29/13	2/5/13	250	249.4	99.756	80-120	
	3A30005-BS1	1/30/13	2/5/13	250	242.2	96.9	80-120	
	3B05009-BS1	2/5/13	2/6/13	250	248.5	99.404	80-120	
	3B05010-BS1	2/5/13	2/6/13	250	250.5	100.208	80-120	
	3B20010-BS1	2/20/13	2/21/13	250	263.8	105.52	80-120	
	3B22012-BS1	2/22/13	2/26/13	250	258.2	103.272	80-120	
	3B26017-BS1	2/26/13	2/27/13	250	254.7	101.888	80-120	
	3B27018-BS1	2/27/13	2/28/13	250	253	101.212	80-120	
	3C01009-BS1	3/1/13	3/4/13	250	268.8	107.528	80-120	
	3C26009-BS1	3/26/13	3/27/13	250	245.8	98.336	80-120	
	3D15004-BS1	4/15/13	4/17/13	250	246.9	98.76	80-120	
	3D16009-BS1	4/16/13	4/17/13	250	259.8	103.94	80-120	
	3D19007-BS1	4/19/13	4/22/13	250	250.3	100.136	80-120	
	3D19008-BS1	4/19/13	4/22/13	250	247.1	98.852	80-120	
	3E14003-BS1	5/14/13	5/15/13	250	232.6	93.036	80-120	
	3E20004-BS1	5/20/13	5/21/13	250	252.9	101.176	80-120	
	3E20005-BS1	5/20/13	5/21/13	250	242.9	97.16	80-120	
	3E21005-BS1	5/21/13	5/22/13	250	245.3	98.116	80-120	
	3E21006-BS1	5/21/13	5/22/13	250	242.4	96.972	80-120	
	3E28011-BS1	5/28/13	5/31/13	250	253.2	101.292	80-120	
	3E29001-BS1	5/29/13	6/3/13	250	237.7	95.064	80-120	
	3F03025-BS1	6/3/13	6/10/13	250	246.3	98.52	80-120	

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Matrices: Water

Client: All Clients

Instruments: All Instruments

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: All Extractions

Arsenic		LCS %R						Limits	Qualifiers
Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R			
	3F11001-BS1	6/11/13	6/12/13	250	223.6	89.46		80-120	
	3F11002-BS1	6/11/13	6/13/13	250	249.2	99.688		80-120	
	3F27001-BS1	6/27/13	7/1/13	250	249.4	99.748		80-120	
	3F27002-BS1	6/27/13	7/1/13	250	249.4	99.76		80-120	
	3F27003-BS1	6/27/13	6/28/13	250	240.7	96.288		80-120	
	3G02004-BS1	7/2/13	7/3/13	250	262.6	105.04		80-120	
	3G02005-BS1	7/2/13	7/3/13	250	269.6	107.84		80-120	
	3G02017-BS1	7/2/13	7/10/13	250	254	101.584		80-120	

Printed: 07/14/2013 07:20

Matrices: Water

Client: All Clients

Instruments: All Instruments

Project: All Projects

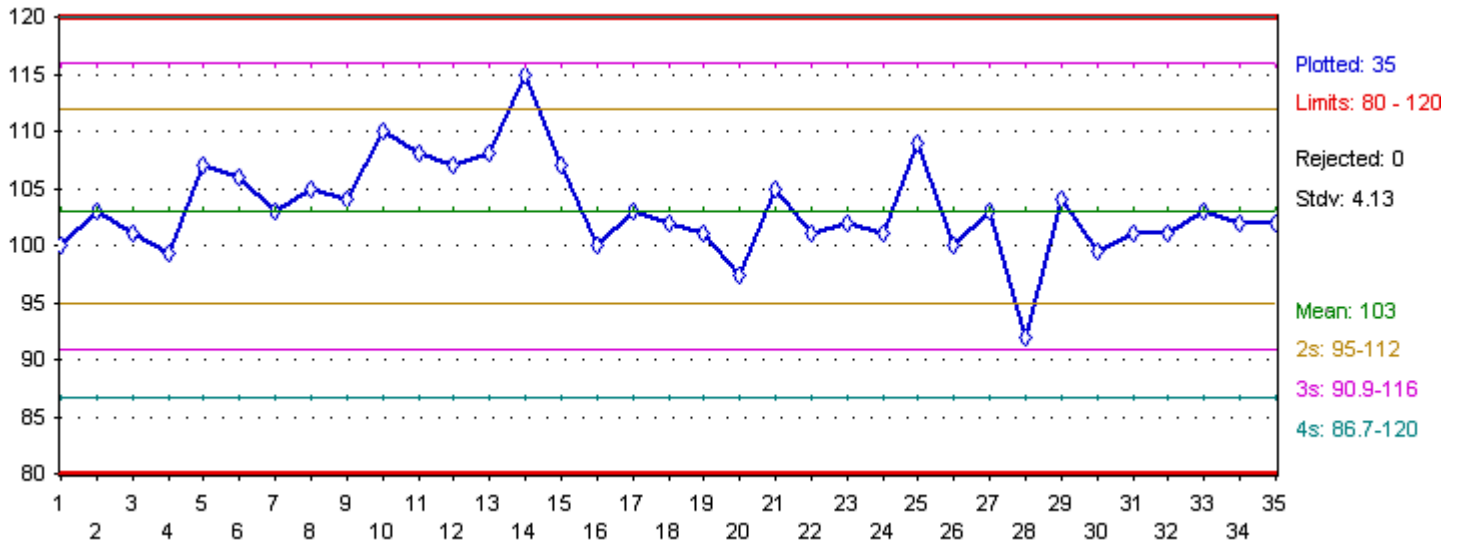
Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: All Extractions

Cadmium

LCS %R



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3A04011-BS1	1/4/13	1/8/13	125	125	99.984	80-120	
	3A16004-BS1	1/16/13	1/18/13	125	128.2	102.584	80-120	
	3A21015-BS1	1/21/13	1/23/13	125	126.4	101.104	80-120	
	3A22016-BS1	1/22/13	1/23/13	125	124.1	99.28	80-120	
	3A29015-BS1	1/29/13	1/30/13	125	133.8	107.08	80-120	
	3A29017-BS1	1/29/13	2/5/13	125	132.7	106.16	80-120	
	3A30005-BS1	1/30/13	2/5/13	125	128.8	103	80-120	
	3B05009-BS1	2/5/13	2/6/13	125	130.7	104.552	80-120	
	3B05010-BS1	2/5/13	2/6/13	125	130.3	104.208	80-120	
	3B20010-BS1	2/20/13	2/21/13	125	137	109.632	80-120	
	3B22012-BS1	2/22/13	2/26/13	125	135	107.968	80-120	
	3B26017-BS1	2/26/13	2/27/13	125	133.6	106.912	80-120	
	3B27018-BS1	2/27/13	2/28/13	125	135.1	108.048	80-120	
	3C01009-BS1	3/1/13	3/4/13	125	144.3	115.448	80-120	
	3C26009-BS1	3/26/13	3/27/13	125	133.8	107.08	80-120	
	3D15004-BS1	4/15/13	4/17/13	125	125.4	100.32	80-120	
	3D16009-BS1	4/16/13	4/17/13	125	128.5	102.792	80-120	
	3D19007-BS1	4/19/13	4/22/13	125	127.8	102.208	80-120	
	3D19008-BS1	4/19/13	4/22/13	125	126	100.776	80-120	
	3E14003-BS1	5/14/13	5/15/13	125	121.7	97.392	80-120	
	3E20004-BS1	5/20/13	5/21/13	125	131.1	104.896	80-120	
	3E20005-BS1	5/20/13	5/21/13	125	126.6	101.288	80-120	
	3E21005-BS1	5/21/13	5/22/13	125	127.3	101.824	80-120	
	3E21006-BS1	5/21/13	5/22/13	125	126.6	101.288	80-120	
	3E28011-BS1	5/28/13	5/31/13	125	136.3	109.064	80-120	
	3E29001-BS1	5/29/13	6/3/13	125	125.5	100.376	80-120	
	3F03025-BS1	6/3/13	6/10/13	125	128.9	103.144	80-120	

Printed: 07/14/2013 07:20

Matrices: Water

Client: All Clients

Instruments: All Instruments

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: All Extractions

Cadmium

LCS %R

Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3F11001-BS1	6/11/13	6/12/13	125	115	92.008	80-120	
	3F11002-BS1	6/11/13	6/13/13	125	130.4	104.328	80-120	
	3F27001-BS1	6/27/13	7/1/13	125	124.2	99.392	80-120	
	3F27002-BS1	6/27/13	7/1/13	125	126.4	101.104	80-120	
	3F27003-BS1	6/27/13	6/28/13	125	126.6	101.312	80-120	
	3G02004-BS1	7/2/13	7/3/13	125	129.2	103.384	80-120	
	3G02005-BS1	7/2/13	7/3/13	125	127	101.632	80-120	
	3G02017-BS1	7/2/13	7/10/13	125	127.6	102.08	80-120	

Printed: 07/14/2013 07:20

Matrices: Water

Client: All Clients

Instruments: All Instruments

Project: All Projects

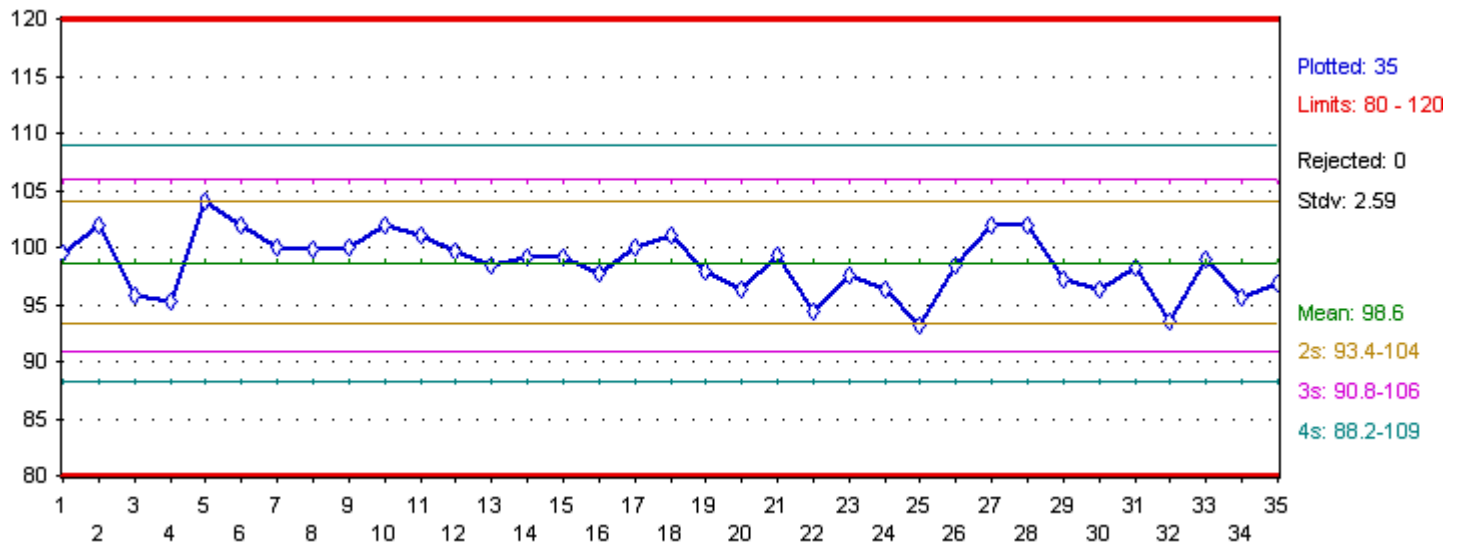
Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: All Extractions

Chromium

LCS %R



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3A04011-BS1	1/4/13	1/8/13	200	198.8	99.42	80-120	
	3A16004-BS1	1/16/13	1/18/13	200	203.4	101.68	80-120	
	3A21015-BS1	1/21/13	1/23/13	200	191.6	95.825	80-120	
	3A22016-BS1	1/22/13	1/23/13	200	190.5	95.235	80-120	
	3A29015-BS1	1/29/13	1/30/13	200	207.1	103.53	80-120	
	3A29017-BS1	1/29/13	2/5/13	200	204.9	102.46	80-120	
	3A30005-BS1	1/30/13	2/5/13	200	200.6	100.3	80-120	
	3B05009-BS1	2/5/13	2/6/13	200	199.7	99.865	80-120	
	3B05010-BS1	2/5/13	2/6/13	200	200.3	100.17	80-120	
	3B20010-BS1	2/20/13	2/21/13	200	203.3	101.665	80-120	
	3B22012-BS1	2/22/13	2/26/13	200	202.1	101.035	80-120	
	3B26017-BS1	2/26/13	2/27/13	200	199.3	99.66	80-120	
	3B27018-BS1	2/27/13	2/28/13	200	197.1	98.545	80-120	
	3C01009-BS1	3/1/13	3/4/13	200	198.2	99.115	80-120	
	3C26009-BS1	3/26/13	3/27/13	200	198.3	99.135	80-120	
	3D15004-BS1	4/15/13	4/17/13	200	195.3	97.655	80-120	
	3D16009-BS1	4/16/13	4/17/13	200	200.2	100.075	80-120	
	3D19007-BS1	4/19/13	4/22/13	200	201.5	100.735	80-120	
	3D19008-BS1	4/19/13	4/22/13	200	195.8	97.91	80-120	
	3E14003-BS1	5/14/13	5/15/13	200	192.7	96.365	80-120	
	3E20004-BS1	5/20/13	5/21/13	200	198.7	99.335	80-120	
	3E20005-BS1	5/20/13	5/21/13	200	188.9	94.445	80-120	
	3E21005-BS1	5/21/13	5/22/13	200	195.2	97.59	80-120	
	3E21006-BS1	5/21/13	5/22/13	200	192.8	96.38	80-120	
	3E28011-BS1	5/28/13	5/31/13	200	186.4	93.215	80-120	
	3E29001-BS1	5/29/13	6/3/13	200	196.7	98.37	80-120	
	3F03025-BS1	6/3/13	6/10/13	200	204.8	102.395	80-120	

Printed: 07/14/2013 07:20

Matrices: Water

Client: All Clients

Instruments: All Instruments

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: All Extractions

Chromium

LCS %R

Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3F11001-BS1	6/11/13	6/12/13	200	204.1	102.035	80-120	
	3F11002-BS1	6/11/13	6/13/13	200	194.4	97.225	80-120	
	3F27001-BS1	6/27/13	7/1/13	200	192.6	96.285	80-120	
	3F27002-BS1	6/27/13	7/1/13	200	196.6	98.325	80-120	
	3F27003-BS1	6/27/13	6/28/13	200	187	93.525	80-120	
	3G02004-BS1	7/2/13	7/3/13	200	197.8	98.915	80-120	
	3G02005-BS1	7/2/13	7/3/13	200	191.5	95.74	80-120	
	3G02017-BS1	7/2/13	7/10/13	200	193.8	96.88	80-120	

Printed: 07/14/2013 07:20

Matrices: Water

Client: All Clients

Instruments: All Instruments

Project: All Projects

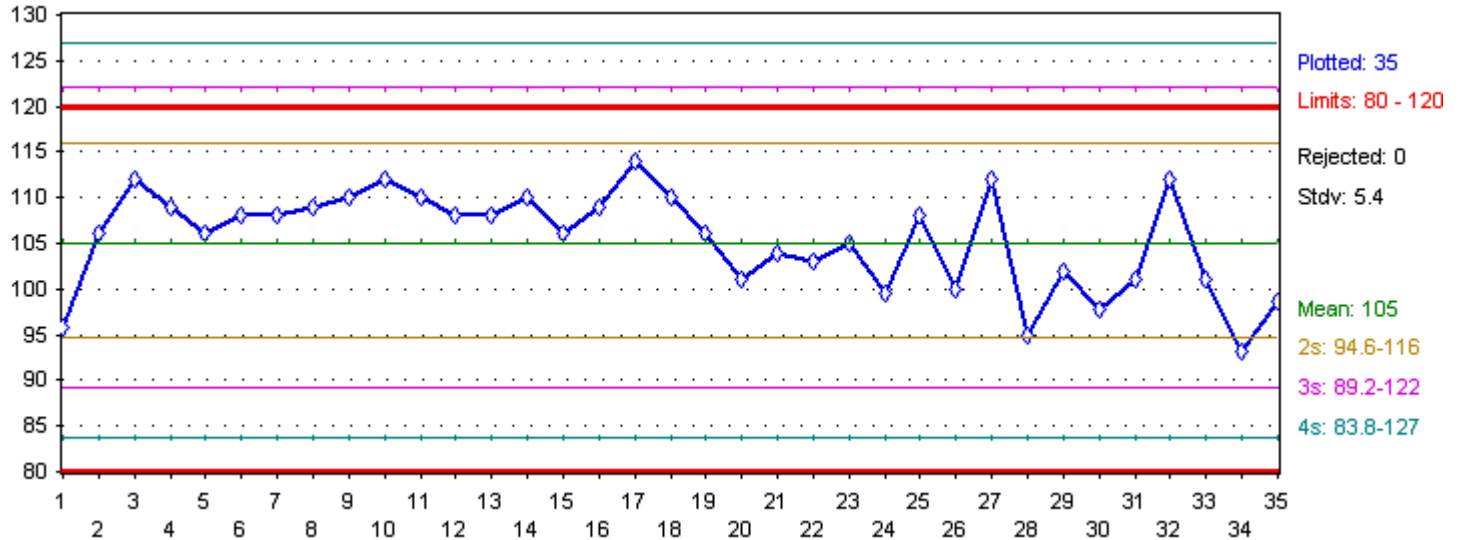
Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: All Extractions

Iron

LCS %R



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3A04011-BS1	1/4/13	1/8/13	1000	957.4	95.744	80-120	
	3A16004-BS1	1/16/13	1/18/13	1000	1062	106.19	80-120	
	3A21015-BS1	1/21/13	1/23/13	1000	1119	111.89	80-120	
	3A22016-BS1	1/22/13	1/23/13	1000	1090	108.99	80-120	
	3A29015-BS1	1/29/13	1/30/13	1000	1061	106.13	80-120	
	3A29017-BS1	1/29/13	2/5/13	1000	1080	108	80-120	
	3A30005-BS1	1/30/13	2/5/13	1000	1080	107.97	80-120	
	3B05009-BS1	2/5/13	2/6/13	1000	1088	108.81	80-120	
	3B05010-BS1	2/5/13	2/6/13	1000	1102	110.24	80-120	
	3B20010-BS1	2/20/13	2/21/13	1000	1116	111.58	80-120	
	3B22012-BS1	2/22/13	2/26/13	1000	1103	110.28	80-120	
	3B26017-BS1	2/26/13	2/27/13	1000	1076	107.56	80-120	
	3B27018-BS1	2/27/13	2/28/13	1000	1077	107.74	80-120	
	3C01009-BS1	3/1/13	3/4/13	1000	1101	110.06	80-120	
	3C26009-BS1	3/26/13	3/27/13	1000	1056	105.64	80-120	
	3D15004-BS1	4/15/13	4/17/13	1000	1086	108.59	80-120	X
	3D16009-BS1	4/16/13	4/17/13	1000	1138	113.79	80-120	
	3D19007-BS1	4/19/13	4/22/13	1000	1102	110.16	80-120	
	3D19008-BS1	4/19/13	4/22/13	1000	1065	106.49	80-120	
	3E14003-BS1	5/14/13	5/15/13	1000	1010	101.02	80-120	
	3E20004-BS1	5/20/13	5/21/13	1000	1044	104.38	80-120	
	3E20005-BS1	5/20/13	5/21/13	1000	1030	103.02	80-120	
	3E21005-BS1	5/21/13	5/22/13	1000	1046	104.56	80-120	
	3E21006-BS1	5/21/13	5/22/13	1000	994.6	99.458	80-120	
	3E28011-BS1	5/28/13	5/31/13	1000	1076	107.6	80-120	
	3E29001-BS1	5/29/13	6/3/13	1000	1004	100.45	80-120	
	3F03025-BS1	6/3/13	6/10/13	1000	1120	112.05	80-120	

Printed: 07/14/2013 07:20

Matrices: Water

Client: All Clients

Instruments: All Instruments

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: All Extractions

<i>Iron</i>		<i>LCS %R</i>					Limits	Qualifiers
Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R		
	3F11001-BS1	6/11/13	6/12/13	1000	949.4	94.936	80-120	
	3F11002-BS1	6/11/13	6/13/13	1000	1018	101.85	80-120	
	3F27001-BS1	6/27/13	7/1/13	1000	978.5	97.848	80-120	
	3F27002-BS1	6/27/13	7/1/13	1000	1007	100.72	80-120	
	3F27003-BS1	6/27/13	6/28/13	1000	1120	112.04	80-120	
	3G02004-BS1	7/2/13	7/3/13	1000	1006	100.58	80-120	
	3G02005-BS1	7/2/13	7/3/13	1000	932.5	93.247	80-120	
	3G02017-BS1	7/2/13	7/10/13	1000	987.1	98.708	80-120	

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Matrices: Water

Client: All Clients

Instruments: All Instruments

Project: All Projects

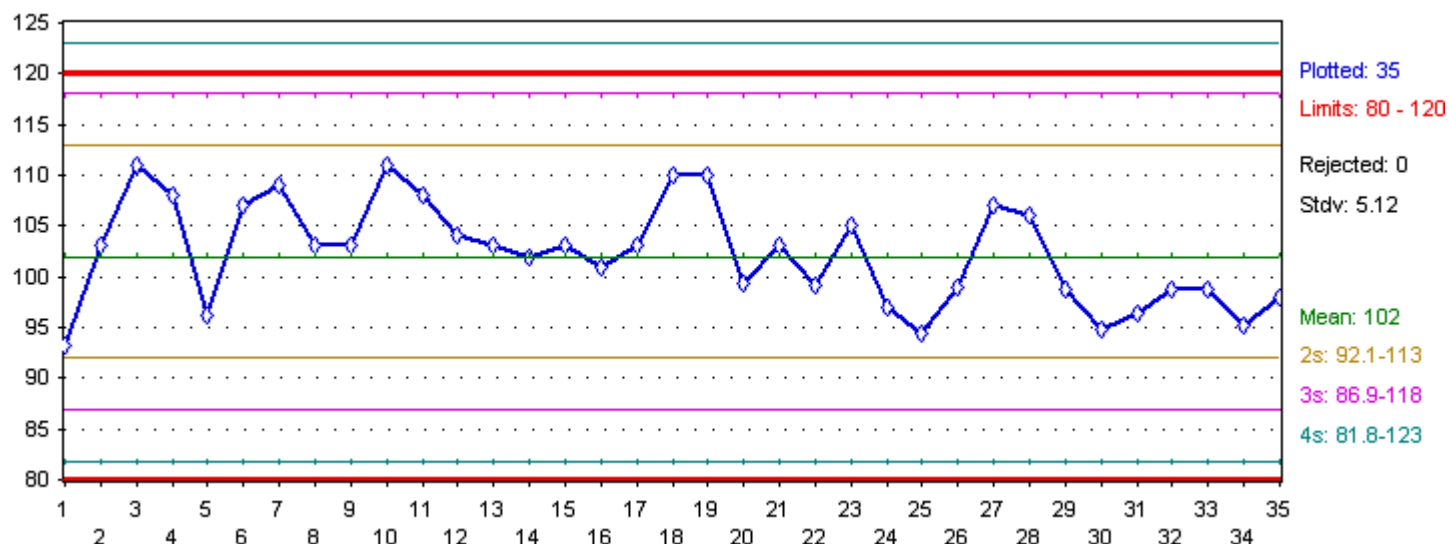
Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: All Extractions

Manganese

LCS %R



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3A04011-BS1	1/4/13	1/8/13	500	466.5	93.306	80-120	
	3A16004-BS1	1/16/13	1/18/13	500	516.4	103.276	80-120	
	3A21015-BS1	1/21/13	1/23/13	500	556.3	111.26	80-120	
	3A22016-BS1	1/22/13	1/23/13	500	541.7	108.346	80-120	
	3A29015-BS1	1/29/13	1/30/13	500	481	96.2	80-120	
	3A29017-BS1	1/29/13	2/5/13	500	536.8	107.352	80-120	
	3A30005-BS1	1/30/13	2/5/13	500	543	108.592	80-120	X
	3B05009-BS1	2/5/13	2/6/13	500	517	103.41	80-120	
	3B05010-BS1	2/5/13	2/6/13	500	516.4	103.278	80-120	
	3B20010-BS1	2/20/13	2/21/13	500	554.6	110.916	80-120	
	3B22012-BS1	2/22/13	2/26/13	500	537.7	107.542	80-120	
	3B26017-BS1	2/26/13	2/27/13	500	519.6	103.93	80-120	
	3B27018-BS1	2/27/13	2/28/13	500	517	103.402	80-120	
	3C01009-BS1	3/1/13	3/4/13	500	509.8	101.968	80-120	
	3C26009-BS1	3/26/13	3/27/13	500	513.3	102.656	80-120	
	3D15004-BS1	4/15/13	4/17/13	500	506.8	101.36	80-120	
	3D16009-BS1	4/16/13	4/17/13	500	514.6	102.922	80-120	
	3D19007-BS1	4/19/13	4/22/13	500	547.7	109.544	80-120	
	3D19008-BS1	4/19/13	4/22/13	500	551.4	110.274	80-120	
	3E14003-BS1	5/14/13	5/15/13	500	496.8	99.36	80-120	
	3E20004-BS1	5/20/13	5/21/13	500	513.5	102.704	80-120	
	3E20005-BS1	5/20/13	5/21/13	500	495.6	99.13	80-120	
	3E21005-BS1	5/21/13	5/22/13	500	524	104.792	80-120	
	3E21006-BS1	5/21/13	5/22/13	500	484.3	96.854	80-120	
	3E28011-BS1	5/28/13	5/31/13	500	472.6	94.53	80-120	
	3E29001-BS1	5/29/13	6/3/13	500	495	99.002	80-120	
	3F03025-BS1	6/3/13	6/10/13	500	536.8	107.364	80-120	

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Matrices: Water

Client: All Clients

Instruments: All Instruments

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: All Extractions

Manganese		LCS %R						Limits	Qualifiers
Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R			
	3F11001-BS1	6/11/13	6/12/13	500	531	106.19		80-120	
	3F11002-BS1	6/11/13	6/13/13	500	494.1	98.828		80-120	
	3F27001-BS1	6/27/13	7/1/13	500	473.9	94.776		80-120	
	3F27002-BS1	6/27/13	7/1/13	500	482.1	96.424		80-120	
	3F27003-BS1	6/27/13	6/28/13	500	494	98.806		80-120	
	3G02004-BS1	7/2/13	7/3/13	500	494.1	98.816		80-120	
	3G02005-BS1	7/2/13	7/3/13	500	475.9	95.178		80-120	
	3G02017-BS1	7/2/13	7/10/13	500	489.3	97.866		80-120	

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Matrices: Water

Client: All Clients

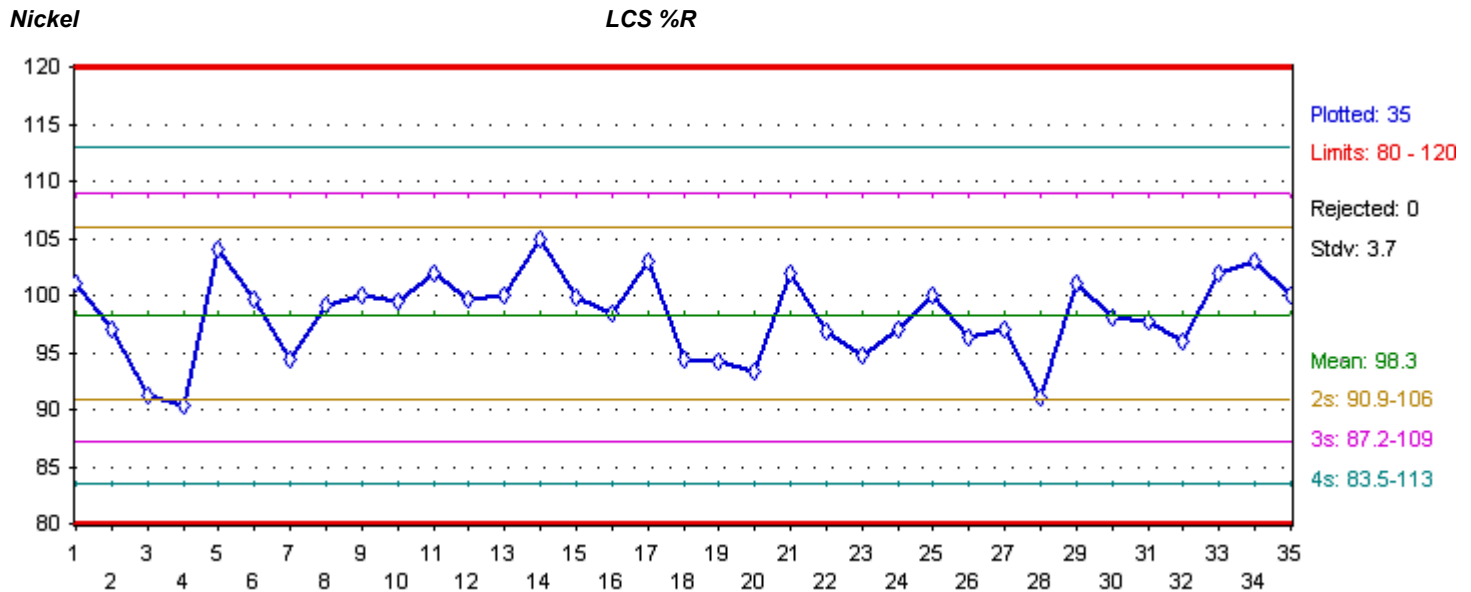
Instruments: All Instruments

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: All Extractions



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	3A04011-BS1	1/4/13	1/8/13	500	506.9	101.376	80-120	
	3A16004-BS1	1/16/13	1/18/13	500	485.7	97.138	80-120	
	3A21015-BS1	1/21/13	1/23/13	500	456.5	91.302	80-120	
	3A22016-BS1	1/22/13	1/23/13	500	452.1	90.428	80-120	
	3A29015-BS1	1/29/13	1/30/13	500	521.8	104.366	80-120	
	3A29017-BS1	1/29/13	2/5/13	500	498.1	99.624	80-120	
	3A30005-BS1	1/30/13	2/5/13	500	471.3	94.254	80-120	
	3B05009-BS1	2/5/13	2/6/13	500	496	99.19	80-120	
	3B05010-BS1	2/5/13	2/6/13	500	501.3	100.262	80-120	
	3B20010-BS1	2/20/13	2/21/13	500	497.3	99.458	80-120	
	3B22012-BS1	2/22/13	2/26/13	500	508.9	101.782	80-120	
	3B26017-BS1	2/26/13	2/27/13	500	498.6	99.73	80-120	
	3B27018-BS1	2/27/13	2/28/13	500	500.8	100.166	80-120	
	3C01009-BS1	3/1/13	3/4/13	500	522.6	104.516	80-120	
	3C26009-BS1	3/26/13	3/27/13	500	499.7	99.936	80-120	
	3D15004-BS1	4/15/13	4/17/13	500	492.7	98.534	80-120	
	3D16009-BS1	4/16/13	4/17/13	500	515.3	103.058	80-120	
	3D19007-BS1	4/19/13	4/22/13	500	471.8	94.368	80-120	
	3D19008-BS1	4/19/13	4/22/13	500	470.9	94.18	80-120	
	3E14003-BS1	5/14/13	5/15/13	500	466.5	93.294	80-120	
	3E20004-BS1	5/20/13	5/21/13	500	508.6	101.72	80-120	
	3E20005-BS1	5/20/13	5/21/13	500	484.1	96.812	80-120	
	3E21005-BS1	5/21/13	5/22/13	500	473.6	94.71	80-120	
	3E21006-BS1	5/21/13	5/22/13	500	485.3	97.058	80-120	
	3E28011-BS1	5/28/13	5/31/13	500	501.3	100.266	80-120	
	3E29001-BS1	5/29/13	6/3/13	500	481.8	96.356	80-120	
	3F03025-BS1	6/3/13	6/10/13	500	484.9	96.974	80-120	

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Matrices: Water

Client: All Clients

Instruments: All Instruments

Project: All Projects

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: All Extractions

<i>Nickel</i>		<i>LCS %R</i>					Limits	Qualifiers
Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R		
	3F11001-BS1	6/11/13	6/12/13	500	455.7	91.134	80-120	
	3F11002-BS1	6/11/13	6/13/13	500	507.4	101.484	80-120	
	3F27001-BS1	6/27/13	7/1/13	500	490.3	98.054	80-120	
	3F27002-BS1	6/27/13	7/1/13	500	488.9	97.772	80-120	
	3F27003-BS1	6/27/13	6/28/13	500	479.7	95.936	80-120	
	3G02004-BS1	7/2/13	7/3/13	500	510.4	102.078	80-120	
	3G02005-BS1	7/2/13	7/3/13	500	514	102.796	80-120	
	3G02017-BS1	7/2/13	7/10/13	500	500.2	100.046	80-120	